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ELECTROFORMING OF ALUMINUM COMPOSITE STRUCTURES BY CODEPOSITION OF HIGH STRENGTH, HIGH MODULUS FIBERS OR WHISKERS

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ABSTRACT

This report describes the task, experiments, developments and studies relating to the optimization of fiber codeposition technology. Process scale-up studies were conducted in the 0.757 m^3 (200 gallon) capacity pilot cell, with the electroforming of a composite structural element, representative of solar array panel section, as an end goal. A laboratory investigation sought to establish codeposition techniques for incorporating high strength, high modulus fibers into an electroformed aluminum composite.

Program objectives were met and all the contract required items were successfully electroformed in the pilot cell. The laboratory investigation concluded that the graphite fiber-aluminum system was the most promising of the various systems studied. These composites exhibited an ultimate tensile strength up to 224 MN/m^2 vs. 76MN/m^2 (32,400 psi vs. 11,000 psi) for plain electroformed aluminum and a modulus of elasticity up to 91.1 GN/m^2 vs. 55.2 GN/m^2 (13.2×10^6 psi vs. 8×10^6 psi) for plain electroformed aluminum. The laboratory process steps were successfully scaled-up in the pilot cell. A graphite fiber-aluminum composite structural element was electroformed in the pilot cell.

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SUMMARY

Contract No. NAS 1-7913 has not only met all immediate contract objectives, but has significantly advanced the metal composite technology by demonstrating the practicality of a novel reinforcing process with wide implications.

The three broad areas of program activities were as follows: (1) Refurbishment of the NASA-Langley owned pilot cell; (2) Development of short length fiber codeposition during aluminum electroforming, to yield reinforced aluminum; and (3) Preparation in the pilot cell of deliverable samples and a fiber-aluminum structural element. The first and the last areas involved more or less routine operation. The bulk of the time and effort went into codeposition studies.

The fiber reinforcement study included over 200 experimental runs in the laboratory aluminum plating cell. Little headway was made during the first five month period. The Technical Program section of this report describes the many potential fiber incorporating techniques investigated and evaluated on the basis of volume fraction attainable, fiber alignment and deposit properties.

Finally, after much experimentation, a method was found to impart sufficient change to the fibers to permit electrophoresis to take place during electroforming. It was found that the fibers aligned during deposition. These breakthroughs occurred late in the contract and the full potential application to this process was not achieved. With this electrophoretic codeposition technique, using graphite fibers, the UTS of aluminum was increased from 76 to 224 MN/m^2 (11,000 up to 32,400 psi) and the elastic modulus from 55.2 to 91.1 GN/m^2 ($8x10^6$ to $13.2x10^6$ psi), though the reproducible values, at this time, are somewhat lower. The bond strength between the aluminum matrix and graphite fibers was approximately 10.4 MN/m^2 (1500 psi), which is a higher than expected value, no doubt due to the absence of any oxide film at the fiber-aluminum interface. The volume fraction achieved at this time is still very low, less than 1%. However, the loading was rapidly increased during the brief period available for the volume fraction investigation. Additional efforts would increase this figure considerably.

Among the many fibers considered in the fiber selection study, graphite was chosen, partly on the basis of its properties and low price and partly on the basis of criteria developed during the program, such as low density, small diameter, conductivity and chargeability, which were necessary for electrophoretic deposition.

INTRODUCTION

Aluminum alloys are used extensively in structures today because of a combination of desirable properties such as: high strength-to-weight ratio, fabricability, availability and low cost. However, the relatively low elastic moduli, 55.2-69.0 GN/m^2 (about 8-10x10^6 psi), have made them less competitive in some structural applications where the stiffness-to-weight ratio is a prime requirement. This and other limitations can be reduced by reinforcing aluminum or its alloys with the new high strength, high modulus, low density fibers now available. The chief advantages of fiber-reinforced aluminum over high strength aluminum alloys include: 1) higher elastic moduli, 2) improved fatigue life and toughness, 3) improved high temperature properties and 4) improved stress rupture life and creep resistance. By controlling the fiber orientation and fiber concentration, anisotropic properties can be imparted to the composite material, which makes it very desirable for structural elements. This also increases the structural efficiency of the reinforced aluminum, because the fibers can be placed so as to provide maximum resistance to multi-axial stresses within the structure.

The key to the utilization of fiber-reinforced aluminum in space applications depends on the successful development of suitable fabrication techniques that produce composite materials and structural shapes, which utilize the properties of the reinforcing fibers. A major problem in aluminum metallurgy results from its ever-present oxide layer, which greatly interferes with the joining and bonding of the aluminum to the fibers and, therefore, makes composite fabrication difficult. Three methods have been used to fabricate aluminum-fiber composites. First is the powder metallurgical process which consists essentially of hot pressing aluminum powders mixed with aligned fibers. However, the resulting specimens have generally been weak, because of damage to the fibers. Second is the plasma spraying technique which involves the spraying of molten aluminum droplets upon fibers that are being wound on a moving mandrel. The temperature of the molten aluminum is relatively low by the time it strikes the fibers, so that degrading chemical interactions are prevented. The plates, thus fabricated, require an additional hot pressing operation to achieve complete densification of the matrix. These composites have exhibited high strengths along the direction of the fibers, although they are relatively weak in the transverse direction. The third process is diffusion bonding, consisting of pressing alternate layers of aluminum sheets and aligned fibers into dense sheets or place composites. High strengths were obtained, although composite shape and fiber configuration attainable is highly limited.

In previous work performed on Contract Numbers NAS 1 - 3319 and NAS 1 - 5743, for the National Aeronautics and Space Administration, Langley Research Center, the aluminum electrodeposition technology was considerably advanced. Aluminum electroforming was scaled-up to commercially significant proportions and a number of 0.76m (30-inch) diameter paraboloidal aluminum electroforms were fabricated. On Contract NAS 1-5743,

the feasibility of codepositing short lengths of silvered glass fibers and aluminum was demonstrated. This room temperature process is not shape limited. Present study sought to extend this technology to high strength, high modulus fibers.

The objectives of the program, sponsored by the National Aeronautics and Space Administration, Langley Research Center, under Contract Number NAS 1 - 7913, were to improve the quality, strength and modulus of electrodeposited composites by using high strength, high modulus fibers and to demonstrate the application of the process to unusual structural shapes applicable to large solar cell arrays. To meet these objectives, the contract set forth specific tasks discussed below.

TECHNICAL PROGRAM

The experimental program, described below, consisted of nine specific tasks directed toward fulfilling the basic contract objectives.

Pilot Cell Refurbishment

Immediate corrective action was required at the start of the program to make the pilot cell suitable for continued electroforming investigation. At the conclusion of the previous contract, it was noted that the plating tank lining was severely degraded. The prime reason for the deterioration (blistering and peeling) of the tank liner was the insurance company's requirement to ground the tank for improved safety. Because of the high ohmic resistance of the ethereal solution and the proximity of both electrodes to the tank wall, the tank itself became a dipolar electrode, evolving gas which tried to push off the lining.

The worn lining was removed and a new fluorinated ethylene propylene plating tank lining was applied. To prevent recurrence of the aforementioned problem, the tank and the cathode were grounded and the wall behind the anode was heavily insulated. Since the tank and the cathode are now electrically connected, both can be maintained at other than ground potential and adjusted as required.

In addition to replacing the defective lining, a specially modified cartridge filter system was installed and several minor design modifications were added, anticipating changing process requirements. The entire system was thoroughly overhauled.

Laboratory Support To Pilot Cell Modification

Laboratory checkout and adjustments of the aluminum plating solution and materials compatability rechecks were performed periodically during the program to insure against any unforeseen problems.

Electroform Flat Pure Aluminum Samples in the Pilot Cell

The refurbished pilot cell was checked out by an initial shake-down run. Subsequently, flat pure electroformed aluminum samples were routinely prepared to fulfill two contractual requirements: - to provide test specimens, for which data are reported in a following section, and to deliver a one square foot electroformed aluminum sheet. This concluded the above task requirements.

Volume Fraction Capabilities in the Pilot Cell

A significant increase in the strength of aluminum deposits with the codeposition of silvered glass fibers was achieved on the previous contract, NAS 1 - 5743, as shown in Table 1. The codeposition mechanism was largely random entrapment of occasional fibers impinging on the surface, due to a temporary arrest on microscopic rough spots. Although fiber volume fraction was not measured under that contract, it is now known that even the "heavier loading" by this method was well below one percent.

The present contract was aimed at improving the quality, strength, and modulus of such composite structures. The contract tasks recognized that improvement of these properties required better control of the deposition mechanism. Increasing the volume fraction of the fibers, their alignment, good bond strength between the fibers and the aluminum matrix, and sound matrix properties are prerequisites to the success, and without these, the substitution of high strength, high modulus fibers for the glass fibers would be meaningless.

With these considerations in mind, a laboratory program was formulated and over 200 laboratory runs, investigating groupings of parameters concurrently, were performed. The development of an improved codeposition technique was the first requirement before pilot cell volume fraction runs could even be considered. A specific high strength fiber for pilot cell study could not be selected at this stage and, primarily because of cost considerations, inexpensive glass fibers were used in the early pilot cell runs.

It was possible to achieve a high (several percent) glass fiber volume fraction by gravity settling, but these composites were porous, basically unsound and limited to flat plates. The use of "levelers" and other additives were examined briefly, although the investigation was directed toward a truly controllable technique of fiber addition to attain the highest fiber volume fraction in a sound composite of complex configuration. Ultimately, the method, found to be the best, was the electrophoretic migration of dispersed fibers to the cathode to enable continuous codeposition with electroformed aluminum. Details are presented in the following section. This method was developed initially with graphite fibers and, with some modification in the pretreatment procedure, it was made applicable to silvered glass fibers. It should be noted, however, that long, 6.35 mm (1/4-inch), length fibers, such as were only avail-

able for glass, are not well suited for electrophoretic codeposition. This is because of a tendency toward increasing fiber "pile-up" with increasing fiber length, and the limited effect of the electrophoretic field on the more massive glass fibers. The upper limit for glass fiber volume loading in a sound composite, attainable in the laboratory, was approximately 1/2%.

The scheduled pilot cell runs with glass fibers offered an excellent opportunity to evaluate scale-up parameters. A glass fiber loading of 0.18% in the laboratory was duplicated (0.20%) in the first pilot cell run, which used the same run parameters (current, frequency of addition, amount of added fibers per amp. hr. of deposition, etc.) as in the laboratory cell. A total loading of 0.30% was attained in the second pilot cell run. Volume per cent was determined by a fiber count for a number of measured cross-sections. Data from these two pilot cell volume fraction runs appear in Figure 1.

A composite specimen from the second pilot cell run was x-rayed to determine the glass fiber distribution throughout the matrix. The photograph, presented in Figure 2, indicated the loading and uniformity of the glass fibers in the aluminum deposit.

Five graphite fiber volume fraction runs were performed to evaluate various codeposition parameters in the pilot cell as applicable to graphite fibers. Included were frequency and quantity of graphite fiber additions to the plating solution, shielding effects, comparison of "straight" plating vs. plating with a periodic reverse cycle.

The fourth run was terminated prematurely (after 7 hours deposition time) when it was noted that the cathode was drawing excessive current. This was subsequently traced to an insulator breakdown in the electrical "feed-through" on the tank, which permitted current leakage to the plating tank. The fifth run duplicated intended conditions of the previous run to evaluate the original parameter objectives. Data from these runs are listed in Table 2.

The increase in size, change in cell geometry, type of agitation and circulation, etc., in the pilot cell, as compared with the laboratory cell in which all the prior work with graphite fibers was performed, created problems which could not be anticipated or solved, except by actual pilot cell operation and subsequent modification of pertinent operating conditions. It was noted that part of the dispersed fibers are "lost" in the large volume, 0.757 m^3 (200 gallons), of plating solution in the pilot cell. Accordingly, the quantity and frequency of fiber additions were increased in the second and third runs. An increased graphite fiber content in the respective composites was noted. However, the low fiber content, even after a ten times "normal" fiber loading, indicated other process modifications were required. It was observed in the laboratory and pilot cell that the lower density (than glass fibers) graphite fibers are quite buoyant, migrate randomly with solution agitation, such that in the large pilot cell the weak electrophoretic field does not provide the predominant moving force.

Therefore, shielding was placed around the cathode to reduce the effect of solution movement, to make the current density more uniform across the cathode and to "trap" fibers in the vicinity of the cathode. The design was a trade-off, since the "ideal" shield would restrict aluminum deposition and virtually eliminate any electrophoretic field acting upon the charged fibers. For comparable graphite fiber additions, the fiber content of shielded composite panels was greater than for an unshielded composite. Greater effective quantities of fibers to the cathode area are required and can be realized by modification of the feed system. Excessive amount of fibers per addition caused fiber pile-up, resulting in considerable "trees" on the deposit surface. The high current density of the "trees" attracted subsequent fiber additions electrophoretically, as well as trapping them physically and, hence, heavier fiber loadings, improperly employed, can be self defeating. Therefore, it is desirable to remove any loose agglomeration of fibers periodically. The cathode shielding used in the pilot cell limited the effectiveness of this removal.

On the basis of these runs, fixture hardware was redesigned several times until in the last run the suction side of the filter pump was modified to act as a "vacuum cleaner" to remove excess, non-adhering graphite fibers from the deposit surface, so that frequent heavy fiber additions could be made without deleterious effects. This device, and other processing improvements, were used in electroforming the 1 sq. ft. graphite fiber-aluminum flat plate (contract deliverable item) described in a later section.

Pertinent data developed during this investigation were (1) that the electrophoretic codeposition process developed in the laboratory was fully applicable to the pilot cell; (2) that the fiber volume fraction attainable in the laboratory was reproducible in the pilot cell; (3) that significant increases in UTS and elastic modulus were achieved with graphite fiber-aluminum codeposits; (4) that some alignment of fibers was achieved (described in detail in the following section); and (5) that the filtration, circulation and cooling systems of the pilot cell make it better suited for attaining sound composites with higher fiber volume fractions than in the laboratory cell.

The amount of fibers added to the 0.757 m^3 (200 gallon) pilot cell solution in each of the five runs amounted only to approximately 2 weight per cent of the aluminum plates which were electroformed. Therefore, even under ideal conditions, the fiber loading had to remain numerically very small, considering that presently undefined portions of the fiber additions were removed by the filter, or were caught on nodules and tree growths, and/or tank walls, or simply remained dispersed in the solution during the 20 hour runs. Therefore, the approximately 10% efficiency of fiber incorporation achieved by the process (i.e., total amount of fibers in the deposit divided by the total fiber addition to the plating cell) is very encouraging. These are rough, average figures and the two significant facts are that (1) the fiber content does increase with the fiber additions, and also that (2) the efficiency figure does increase with better shielding. Both facts point toward fruitful areas for further development.

High Strength, High Modulus Fiber-Aluminum Composites

The feasibility of incorporating fibers into electroformed aluminum deposits had previously been established during an in-house program. This technique utilized short length fibers dispersed in the ether-solvent aluminum plating solution and yielded, by codeposition, an increased strength electrodeposit. Work on Contract NAS 1 - 5743 improved the mechanical properties of electroformed aluminum and extended the electroforming process technology. Short length glass fibers were employed for that development study, since it was readily available, inexpensive and well suited for experimentation. However, it was realized that glass fibers in a composite would not yield high absolute values of strength and would not increase the elastic modulus, but this selection was justified, since the process developed for the incorporation of glass fibers could be modified later for other reinforcing materials. Contract NAS 1-7913 sought to advance this novel codeposition technology and was directed primarily to the high strength, high modulus fibers.

For clarity of presentation, the following sections are reported sequentially and individually, although the actual parameters were studied concurrently, since there was considerable interaction between them.

Selection of Fibers: The selection of short fibers to be used as reinforcing agents for this program was based on several considerations, which are summarized in Table 3. On these bases, it was decided to concentrate this development effort on three fiber types: (1) graphite fibers, (2) Al_2O_3 whiskers and (3) SiC whiskers. However, the major effort has been with the graphite fibers. The reasons for the use of and emphasis on these fibers are discussed below.

Since 1959, a variety of new reinforcing fibers have become available. The higher elastic moduli of new fibers, compared to that for glass fibers, has evoked considerable interest in their utilization. Structural elements, reinforced with these new fibers, should exhibit 5 to 10 times greater stiffness than comparable glass filament reinforced structures. Furthermore, the new fibers have many other desirable features: lower sensitivity to mechanical damage, greater corrosion resistance, higher strength retention at elevated temperatures and, for some, lower density than glass (i.e., 1661 Kg/m^3 or 0.06 $lb/in.^3$ vs. 2491 Kg/m^3 or 0.09 $lb/in.^3$ for graphite and glass fibers, respectively). The properties and potential of these fibers have been reported in detail (Ref. 1, 2, 3 and 4). Some of the mechanical properties of the fibers of interest are compared in Table 4. These include the fibers formed by chemical vapor deposition on hot filamentary substrates, such as boron deposited on fine tungsten wires (B/W), boron deposited on fused silica (B/SiO_2) etc., the glass fibers, the graphite fibers, which have about the same diameter as the glass fibers, and the single crystal fibers, or whiskers.

Figure 3A compares the strength and strength-to-weight-ratios (specific strengths) of several fibers. Only the tensile strength for the whiskers

exceeds that for the glass filaments. On a strength-to-weight basis, the values for the other fibers are nearly equal to that for glass. However, on the basis of elastic modulus, or specific modulus, Figure 3B shows that all of the newer fibers are far superior to the glass fibers. In terms of specific modulus, one of the graphite fibers (RAE-60) exceeds that for all the other fibers.

The graphite fibers are small in diameter (about 0.3 mils). This is desirable, since even in a random fashion, they can be packed more closely together than the larger, vapor deposited fibers, which are 4 mils in diameter. Thus, the inter-fiber spacing of the B/W type fibers would be much greater, and would present some problems in filling these voids completely with aluminum, as illustrated in Figure 4. The larger fibers would tend to be coated individually first before the aluminum would plate the interstices. The finer fibers (0.3 mil diameter) could be codeposited and incorporated more readily as the aluminum layers are built-up from the substrate or mandrel.

The graphite fibers are electrically conducting and, therefore, do not require a prior application of conductive coatings. However, some types of graphite fibers have a surface coating of a PVA sizing, which must be removed prior to electroplating. In terms of cost, most graphite fibers are less expensive than all the other fiber types listed except for glass. Also, these fibers are now commercially available in quantities of hundreds of pounds, and in another year or two they should be available by the ton.

One of the chief advantages of these new fibers is their extremely high specific modulus. Thus, they should impart significant stiffness to the aluminum. They should also reduce the density of aluminum. When considering these factors and other criteria (as listed in Table 5), the RAE-60 graphite fibers appeared very promising for this study. The more dense and costly Al_2O_3 and SiC whiskers were also attractive fiber reinforcement candidates for preliminary studies. Although the Al_2O_3 whiskers are not electrically conductive, they are available in the coated form.

In summary, the graphite fibers were the most attractive fibers which could be used on this program from the standpoint of desirable properties, size (length and diameter), cost, and availability. Similarly, the Al_2O_3 and SiC whiskers were also attractive candidates, although considerably more expensive. Thus, the emphasis was directed primarily to the graphite fibers.

Fiber-Aluminum Codeposition - Over 200 laboratory runs, investigating pertinent codeposition parameters, were made. Eighteen codeposition laboratory runs in a nickel sulfamate bath preceded the work in the laboratory aluminum plating cell. Nickel sulfamate screening runs reduced the workload in the heavily occupied aluminum plating glove box and enabled visual observation of the codeposition mechanisms. This approach was acceptable, since many basic electrodeposition parameters do not depend on the particular system involved. Still, where warranted, confirmatory runs were made in the aluminum plating cell.

The first process investigated in the nickel cell was electrophoretic codeposition. In this earlier novel process (GE pat. Docket No. 39-1D-985), we found conditions to codeposit solid particles by simultaneous electrophoresis with metal deposition. The amount co-deposited is well controllable. While this process works well with powders or spherical particles, it was initially unsuccessful with the fibers. By repeated reduction of the glass fiber length, we found that at greater than a 10:1 length to diameter ratio, the electrophoretic transport of fibers within the solution became negligible. Further work, described in a later section, enabled using greater length fibers. This was verified in the aluminum solution with both glass and with graphite fibers, though conditions in the higher ohmic resistance aluminum bath should have favored electrophoretic transport in comparison with the nickel sulfamate bath. In this investigation, we were aiming at minimum length to diameter ratios of 100, preferably 1000.

Repeated runs with increased fiber content in the plating bath only confirmed the fact that random occlusion will not significantly increase the fiber volume fraction in the deposit. The minimum loading obtainable did not approach the fiber volume fraction aimed at in this study.

A procedure for inclusion of graphite fibers by "tamping" was investigated. Prior to the start of the aluminum deposition process, the substrates were routinely activated in a 15% oleic acid bath to remove surface contaminations, including thin oxide films. Since this oleic acid film was sticky, the surface was very receptive to an initial short length fiber addition. After sprinkling or blowing fibers over the sticky surface for some runs, additional rollering or tamping flattened down the fibers. However, the vigorous reaction between the bath and the oleic acid film lifted away many fibers from the substrate, thereby limiting the effectiveness of the method.

In further refinements of the lay-up process, the oleic acid film containing the fibers was covered with (1) glass cloth (as shown in Figure 5.), (2) glass filter "paper" (see Figure 6.), or (3) perforated polytetrafluoroethylene film (1 mil), illustrated in Figure 7. All of these worked well in retaining and permitting encapsulation of the fibers. However, by the time each could be removed (1/2 hour), this cover was firmly locked into the aluminum deposit. Since these were unplanned reinforcing materials, this was unacceptable. Earlier removal did not assure retention of all the fibers, since some of the fibers still tore off with the retention material. This is shown in Figure 8.

Finally, a 1/32" thick, perforated (1/8" diameter perforations) unplasticized PVC overlay, which was used, appeared the best suited (see Figure 9.). The aluminum deposit could not grab the comparatively thick PVC overlay within the 1/2 hour necessary for the retention of the fibers. Several runs established the pressure necessary for holding down the fibers.

Since the temporary retention of fibers by the perforated unplasticized PVC overlay yielded encouraging results, thoughts were given to additional layers of fibers beyond the initial one. A procedure was worked out to assure good adhesion of the subsequent aluminum deposit to the previously deposited aluminum surface. The most successful procedure required oleic acid activation of the clean surface, followed by a 150 a.s.f. reverse current treatment in the plating solution prior to the start of the deposition. This procedure permitted removal of the initial fiber-containing deposit from the bath as soon as the deposit thickness exceeded the fiber diameter, and a subsequent aluminum build-up after the application of more fibers to the surface.

Although the alignment and the volume fraction of fibers were fairly well controllable by this technique, it was not strictly a codeposition process and, therefore, less desirable for meeting the objectives of the present contract.

Dielectrophoresis was tried as a procedure for depositing fibers directly on the cathode. Dielectrophoresis can be defined as the translational motion of a neutral particle due to the action of an inhomogeneous electric field on its permanent or induced dipole moment. This is distinguished from the motion caused by the response to a free charge on a body in a homogeneous electric field (i.e., electrophoresis).

To create a non-uniform field, a large plate anode and a small cylindrical cathode (heavy wire) were introduced into the aluminum plating solution-fiber dispersion. Migration of the dispersed graphite fibers to the cathode (high current density pole) was observed. No fibers migrated to the anode. The deposited fibers were tangentially aligned (perpendicular to the rod axis). This investigation looked highly promising and a larger cylinder could have served as the cathode in the scaled-up 0.757 m^3 (200 gallon) plating solution. However, with the sudden advance made by the electrophoretic method, described below, the dielectrophoretic investigation was shelved.

Electrophoretic migration of high strength, high modulus fibers to the cathode during aluminum electrodeposition represented, potentially, an ideal method for achieving "high" fiber volume inclusion and fiber directionality. Difficulties in utilizing this technique occurred because of a high fiber length-to-diameter ratio initial requirement in the electroform. Reconsidering the defined program goals - to extend the state-of-the-art of the aluminum codeposition process, in order to increase composite UTS and modulus without immediately aiming at the maximum possible values - substantially lower fiber length-to-diameter ratios could be untilized to meet these objectives. Although the opportunity to attain theoretical strength was diminished, it was anticipated that the practicality of working with short length graphite fibers would compensate for this.

Dispersions of short length, 0.8 mm (~1/32-inch), graphite fibers, with a length-to-diameter ratio of approximately 100, were prepared in

a number of solvents, prior to introduction into the aluminum plating solution. Electrophoretic migration of the graphite fibers to the anode occurred; however, a like movement to the cathode is required to fabricate aluminum composites. Many dispersants and various charging procedures were tried, in vain, to alter the electrical charge of the fibers. Consultants, in the fields of colloidal dispersion and electrophoretic kinetics, within the many departments of the company and various outside companies and agencies, were contacted. The consensus of opinion was that fiber pretreatment was necessary, but that it would be impossible to specify any dispersant which would give, predictably, the desired electrical charge to the fibers in our non-aqueous plating system. A trial and error approach was recommended.

The right combination, to deposit the graphite fibers on the cathode, was finally found after weeks of experimentation. Short length graphite fibers were ultrasonically dispersed into ether, which contained an anionic dispersant. Addition of the fibers to the aluminum plating bath, prior to or during plating, resulted in heavy fiber migration to the cathode. The initial investigation was qualitative and an overly heavy loading was achieved in the deposit. The system looked very promising and the laboratory investigation concentrated on methods for controlling the migration rate.

Additional work, requiring many laboratory test runs, was performed, and procedures for maintaining suitable control of the fiber migration rate, necessary to achieve sound, uniformly distributed fiber-aluminum composites, were developed.

The deposition on the cathode occurred uniformly and in one plane, without any pile-up or bunching of fibers. The similarly charged fibers repelled one another sufficiently, even after deposition on the cathode, so that fiber pile-up was not observed, except when the fiber addition was excessive. This is extremely important, since fiber-to-fiber contact is a weakening factor in the fabrication of composites.

Inherent contract limitations and the necessity of evaluating all of the many promising codeposition methods earlier in the program, severely limited the time for optimization of this promising technique. Many vital parameters still require additional study; however, much important data have been amassed (reported in a later section).

A number of other dispersing techniques were investigated prior to, or concurrently with, the method described above. These efforts will be described, briefly.

An interesting approach was developed by the Lincoln Laboratory in Massachusetts, for giving the graphite fibers a positive charge. This consisted of electrically charging dry air by passing it between condenser plates, separating one charged stream, and passing it, turbulently, through a batch of graphite fibers. In this manner, a static charge was transferred to the fibers. An apparatus for electrically charging air was

constructed. Unfortunately, the static charge quickly dissipated when the fibers were introduced into the plating solution.

Other techniques, which were investigated, were to degas the graphite fibers and either (1) apply an inert gas film (i.e., N_2); or (2) apply a reducing gas film (i.e., H_2), or (3) apply an oxidizing gas film (i.e., O_2), or (4) transfer the fibers immediately into the plating solution or (5) apply a polar compound, trial and error method, until the desired action in an electrophoretic field is attained. However, with the electrophoretic procedure improvement described previously, the investigation of these techniques was abandoned.

Physical Properties of Laboratory-Prepared Codeposits - Early laboratory-prepared codeposits, particularly by the tamping procedure, showed decreases in ultimate tensile strength. Graphite fibers codeposited electrophoretically with the aluminum matrix yielded specimens in which a marked increase in strength was noted. Operating conditions were varied to investigate pertinent parameters, and subsequently some samples tested higher, while some tested lower. Supposed like specimens did not always yield identical test results, which indicated that proper control and understanding of controlling parameters had not yet been realized.

Accordingly, some graphite fiber-aluminum composites have shown significant increases in ultimate tensile strength - up to 224 MN/m^2 (32,400 psi) vs. 76.0 MN/m^2 (11,000 psi) for plain electroformed aluminum. Some graphite fiber-aluminum composite samples have shown a marked increase in the modulus of elasticity - up to 91.1 GN/m^2 (13.2 x 10^6 psi) vs. 56.6 GN/m^2 (8.2 x 10^6 psi) for plain electroformed aluminum. The plain electroformed aluminum control specimens were not identified as such to the testing laboratory, and at least one such control specimen was included with each batch of specimens and tested concurrently. These control samples were machined to the same dimensions as the test specimens and by appearance were undistinguishable from them. The test procedure, established prior to the start of this program, embodies the mounting of two strain gages on each specimen - one on each face of the gage length. The stress-strain curve, subsequently obtained, averaged the two individual curves and eliminated any extraneous effects.

These increased values, cited above, cannot be explained by the rule of mixtures theory of fiber reinforced composites, because of the low volume per cent of contained graphite fibers (approximately 1%). Therefore, the mechanism of strengthening meritted some investigation.

An electroformed aluminum specimen prepared from a new laboratory plating solution, and a graphite fiber-electroformed aluminum codeposited specimen, prepared from an identical plating solution, were metallographically polished and etched with HF. Photomicrographs of the two materials revealed that there was no promounced pattern in the plain electroformed aluminum, but an intricate pattern of fine lines was observed in the microstructure of the reinforced material. This pattern is shown in Figures 10 and 11, a transverse section and longitudinal section, respectively, of the

graphite fiber-aluminum specimen. The line pattern appears to be anisotropic and may be related to the arrangement of the electrodes and the resulting electromagnetic field patterns. The potential significance of this will be discussed in the following section.

These fine lines in the microstructure appear to be microcracks. Such cracks are evidence of high internal strains developing during the co-deposition electroforming process. Such high strains induce dislocations, strain hardening and, consequently, an increase in UTS. The origin of these strains may be due to adsorbed materials, from the surfaces of the graphite (to induce the proper charge for migration to the cathode), entering into the electrodeposition process. An important characteristic of such effects is the low threshold of impurity concentration needed to produce significant changes in the morphology of an electrolytically growing crystal.

In addition to the effect on UTS of internal strains, which would not effect modulus of elasticity, the graphite fibers, per se, may be reinforcing even though present in a very small volume percentage. For example, Mantei (Reference 5) has shown that a single boron fiber in aluminum (less than 1 volume per cent) can induce significant increases in ultimate tensile strength and modulus of elasticity. These increases exceed predicted values from the rule of mixtures theory. A basic assumption of this theory is that Poisson's ratio for the matrix and for the fiber be identical. This is not so for graphite fibers in aluminum, where the graphite fiber Poisson ratio is considerably lower than that for aluminum. As a result, a state of triaxial stress is induced during tensile test (References 6 and 7) and this reduces the possibility of slip and yield and, thereby, tends to increase the UTS. According to Mantei's experiments, some synergistic effect also increased the modulus of elasticity. Similar data on synergistic effects of fiber-matrix composites strength and modulus have been reported (References 5, 6, 7, 8 and 9).

In the case of the electroformed composite, other factors, which may be operating to increase UTS and E even with low volume fraction loadings of fibers, are texture effects (Reference 10), preferred crystallographic orientation, grain refinements due to specific additives used and impurity atoms in the lattice.

The following items deserve special consideration:

(1) The standard value for modulus of elasticity of electroformed aluminum is 56.6 GN/m^2 (8.2 x 10^6 psi), as determined on several dozen test samples over the past four years. This is less than the book value for other types of fabricated aluminum. However, a similar circumstance exists for nickel electroformed from a "low" stress nickel sulfamate plating solution - 124 to 152 GM/m^2 (18 to 22 x 10^6 psi) vs. 207 GN/m^2 (30x10^6 psi) for "conventionally" fabricated nickel (Reference 11).

(2) High UTS and E values have been reproduced, although not on a consistant basis. Modulus increases have occurred only in about 1/3 of

the tested specimens. However, this frequency attests to significant improvement, even if to inadequate control and insufficient definition of the applicable parameters.

(3) Data variations are larger in the small lab cells, where a few hours run causes marked changes in the composition of the small quantity of plating solution. Also insufficient control exists over other parameters, such as the effect of length to diameter ratio of the "short", handcut fiber lengths, fiber dispersant chemicals, degree of alignment, actual fiber loading and uniformity and the like.

(4) Additional efforts in the graphite-aluminum codeposition method are required for this recently established system and technique.

Fiber-Alignment - The metallographic evidence of anisotrophy of the deposited graphite fibers, previously supposed random in one plane, led to taking specimens with the sample axis cut parallel to the anodes and with the sample axis cut perpendicular to the anode position during electrodeposition. The modulus of elasticity of tensile specimens cut in the transverse direction exceeded that for the longitudinally cut counterpart - 72.5 GN/m^2 (10.5 x 10^6 psi) vs. 58.0 GN/m^2 (8.4 x 10^6 psi). The data are very limited, at this time, but significant, considering they substantiate the metallographic work. The importance of aligning fibers by controlling the directionality of the electromagnetic field cannot be overemphasized. The time remaining on the contract did not permit pursuing this promising development any further. The technique of aligning fibers by controlling the directionality of the electromagnetic field is a major part of any planned future investigation.

Earlier in the program, another technique for achieving fiber alignment was investigated. This method, termed solution streaming, required continuous non-turbulent transport of a quantity of plating solution containing dispersed fibers over the cathode. It was hoped that the flow velocity would exert a positive force for alignment, but this proved inadequate. One possible solution conceived was to coat the graphite fibers with nickel and impose a magnetic field to align the fibers. A number of variations on this basic approach appeared very promising, although they were not followed up in detail, since the electrophoretic alignment was more universally applicable. The streaming method is described in detail in Appendix A.

Fiber-Matrix Bond Strength - The increased strength and modulus values measured for various graphite fibers-aluminum codeposits represented an indirect qualitative indication of fiber-to-matrix bond strength. The need for obtaining a quantitative determination existed, since carbon and aluminum are inert with respect to one another below 1073°K (800°C) and since a fiber reinforcement would not be effective if a weak bond strength condition existed.

A limited investigation of the graphite fiber-to-electrodeposited aluminum bond strength was undertaken. A number of individual 7.6-micron (0.0003-inch) diameter graphite fibers were affixed to a frame-like rack, after prior fiber cleaning by soaking in alcohol. Subsequently, a partial length of each fiber was electroplated with aluminum. The deposit on each fiber was spotty and discontinuous as typified by an improperly cleaned surface. Several additional racks were prepared using fibers previously cleaned by boiling in water - our usual procedure. The aluminum subsequently deposited was continuous and comparable to the quality on codeposited fibers.

The test procedure consisted of attaching the short section of deposited aluminum of an individual filament to an arbor with epoxy cement and subsequently pulling the fiber in tension in a micro-tensile testing machine. To measure the bond strength, it was necessary to "pull out" the graphite fiber from the deposited aluminum sleeve. The "long" continuous length of aluminum on the graphite fibers presented a problem, in that the fibers themselves failed prior to "pull out". Short lengths of deposited aluminum were required. Strange to report, the spotty discontinuous aluminum deposit was best suited for testing. These short aluminum nodules were of various lengths and in the range suitable for "pull out" of the fiber from the aluminum matrix. Shorter and shorter lengths were used in subsequent tests until finally a fiber "pull out" was achieved for a 0.13 mm (0.005-inch) length of aluminum. A bond strength of 10.4 MN/m^2 (1510 psi) was calculated from the data. A detailed write-up of the procedure, calculations, etc., appears in Appendix B. It was concluded, from this investigation, that the bond strength between the graphite fiber and electrodeposited aluminum is not a limiting problem for obtaining composite reinforcement at room temperature. Also, the short length fibers utilized for codeposition exceed the critical fiber length.

It should be noted that the graphite fiber was used substantially in the as-received condition and, to date, these fibers have not been surface treated, etc., to attempt to increase bond strength. This is clearly a very fertile area for further study.

Electroform Flat Glass Fiber-Aluminum Samples in Pilot Cell

Although the limitations (described earlier) of glass fiber codeposition by electrophoresis was recognized, the electroforming of glass fiber-aluminum samples was deemed important as a dress rehearsal for graphite fiber run (described in the following section) and to provide reference specimens for test.

Glass fibers were silvered according to techniques previously reported (Reference 12). Subsequently, flat glass fiber-aluminum samples were routinely prepared to fulfill contractual requirements - to provide test specimens, for which data are reported in a following section, and one square foot of glass fiber-electroformed aluminum sheet, a contract de-

liverable item. This concluded the above task requirements.

Electroform Flat Graphite Fiber-Aluminum Samples in the Pilot Cell

Preparations were made for electroforming a graphite fiber-aluminum composite in the pilot cell utilizing our projected thinking on process optimization, incorporating such features as the "vacuum cleaner" (described earlier) for removing excess, non-adhering fibers. This does not represent the furthest possible advance, but does mark a considerable step forward in this new codeposition technology.

Flat graphite fiber-aluminum samples were routinely prepared to fulfill contractual requirements - to provide test specimens, for which data are reported in the following section, and one square foot of graphite fiber-electroformed aluminum sheet, a contract deliverable item. This concluded the above task requirements.

Physical Properties of Plain, Glass Fiber & Graphite Fiber-Aluminum

Specimens of plain aluminum, of glass fiber-aluminum and of graphite fiber-aluminum composites were tested, as described previously. These specimens were taken from locations adjacent to each of the three contract deliverable panels. Transverse and longitudinal specimens were selected for the graphite fiber-aluminum composite.

The glass fiber-aluminum specimen exhibited little increase in strength. An ultimate tensile strength and modulus of elasticity of 184 MN/m^2 (26,700 psi) and 64.9 GN/m^2 (9.4 x 10^6 psi). respectively, for graphite fiber-electroformed aluminum was measured, as compared with an average of 97.4 MN/m^2 (14,100 psi) and 48.3 GN/m^2 (7 x 10^6 psi), respectively, for plain electroformed aluminum. These data are presented in Table 6. Representative stress-strain curves for each specimen are shown in Figures 12, 13 and 14.

Thermal expansion of the three specimens were 11.0/°F, 11.2/°F and 11.4/°F for plain aluminum, glass fiber-aluminum and graphite fiber-aluminum, respectively.

The measured fiber volumes were 0.15% and 0.11% for glass fiber and for graphite fiber-aluminum composites, respectively.

Electroform Graphite Fiber-Aluminum Structural Element

Seven designs for electroforming the graphite fiber-aluminum codeposition composite structural element in the pilot cell were proposed. The applicability of each proposed design, relating to specific structural and configuration usefulness in solar panel design, was discussed in detail with Mr. Ken Hanson, of GE's Space Power Group. Subsequent discussions with NASA-Langley technical personnel established the preferred structural element design, a panel section with upper tubular support. Accordingly, a scaled-down version mandrel (shown in Figure 15) was fabricated for a "shake-down" run to establish

the current profile, etc., for that specific geometry. Subsequently, an increased size, to scale, brass and copper mandrel was fabricated and a graphite fiber-aluminum composite was codeposited thereon in the pilot cell. The mandrel was etched away, leaving the required free-standing structural element, appearing in Figure 16.

CONCLUSIONS AND RECOMMENDATIONS

NAS 1-7913 was a highly successful contract. The immediate objectives of improving the quality, strength and elastic modulus of electrodeposited composites, utilizing high strength, high modulus fibers and of demonstrating the applicability of the electrodeposition process to unusual structural shapes, as might be used in large solar cell arrays, was clearly met. In addition, considerable advances in the overall technology were made, particularly considering the real breakthroughs that occurred in the last 1½ months of the laboratory study. Highlights may be summarized as follows:

1. The practicality of aluminum matrix composite fabrication by electroforming was demonstrated.

2. Short length, dispersed fibers were electrophoretically co-deposited in a controlled manner.

3. Both the strength and elastic modulus of aluminum were increased significantly after the codeposition of fibers. The UTS and elastic modulus were increased up to 300% and 160%, respectively, of the base material.

4. The above noted increases occurred in composites with less than 1% fiber content. Only at the end of the program were methods found which permitted continued increases in the fiber loading, as compared with the negligible amounts added initially.

5. The mechanism of this reinforcement is not evident. However, these data were reproducible and the values increased with greater fiber loading within the limited volume fraction investigated to date.

6. Metallographic examination, later confirmed by mechanical test, indicated that orientation of the fibers within the composite occurred, due to the non-uniform directionality of the electric field.

7. A good graphite fiber-to-aluminum matrix bond strength was measured, particularly when considering that no surface pretreatment was utilized.

The data generated on Contract No. NAS 1-7913 indicate that considerable advances in this new technology were made and that we are on the threshold of a truly significant breakthrough in this room temperature, pressureless fabrication procedure. Therefore, continuation of the program efforts

are highly recommended. This new work would be directed toward optimization of the techniques for fabricating fiber-aluminum composites by codeposition, concentrating on increasing fiber-matrix bond strength and fiber loading, and improving and controlling fiber alignment. The basic mechanism and areas for investigation have been established on this contract. The rewards and big payoff await further development.

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10. Telephone communication with Dr. William Stuhrke, formerly with Wright Patterson Field, describing work performed by the University of Tennessee and sponsored by Wright Field.

11. Electroforming brochure, GAR Precision Parts, Inc., Stamford, Conn.

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T A B L E 1.

EFFECT OF FIBER-INCORPORATION INTO ALUMINUM ELECTROFORMS[1]

		Glass Fiber-Incorporated Electroformed Aluminum				
	Pure Electroformed Aluminum	Light Loading	Heavier Loading 20 asf	Heavier Loading 30 asf	Increased Loading	Improved Processing
Ultimate Tensile Strength MN/m^2	76.3	76.3	98.9	87.2	113.	143.
(psi)	(11,050)	(11,050)	(14,320)	(12,625)	(16,296)	(20,700)
Yield Point @0.2% Offset, MN/m^2	583					126.
(psi)	(8,450)	--	--	--	--	(18,300)
Elongation, %	26	(2)	(3)	11.0	12	(2)

(1) Work performed on Contract No. NAS 1-5743

(2) Not Measured

(3) Specimen broke in vise grip and could not be measured.

T A B L E 2.

Graphite Fiber Volume Fraction Data from Pilot Cell Runs

RUN NO.	Graphite Fiber Volume Per Cent	UTS MN/m^2	UTS ksi	Elastic Modulus GN/m^2	Elastic Modulus $x10^6$ psi	OPERATING CONDITIONS: Straight Plating or Periodic Reverse	OPERATING CONDITIONS: Fiber Added (grams)	OPERATING CONDITIONS: Shielding or Cathode	Remarks
1.	0.024	107(a)	15.5(a)	67.2(a)	9.73(a)	Straight Plating	0.5	No	None
2.	0.071	104(a)	15.1(a)	62.1(a)	9.0(a)	" "	2.0	No	None
3.	0.114	112(a)	16.2(a)	65.9(a)	9.55(a)	" "	4.0	No	None
4.	0.090	127(a)	18.4(a)	62.5(a)	9.05(a)	Periodic reverse	2.0	Yes	Breakdown of electrical feed through insulator necessitated aborting run.
5.	0.114	108(a)	15.6(a)	58.0(a)	8.4(a)	Periodic reverse	2.0	Yes	Repeat of above run.
		117(b)	16.9(b)	72.5(b)	10.5(b)				

(a) Tensile specimens cut in longitudinal direction.

(b) Tensile specimens cut in transverse direction.

TABLE 3.

CONSIDERATIONS FOR SELECTING A SPECIFIC REINFORCING FIBER

A. MECHANICAL PROPERTIES

1. Strength (and strength scatter)
2. Elastic modulus
3. Density
4. Size
   - (a) Length - continuous
     - short
   - (b) Diameter
5. Shape (cross-section, taper, kinked, etc.).
6. Surface properties
7. Hardness

B. AVAILABILITY

1. Cost
2. Form
   - (a) Monofilament
   - (b) Staple
   - (c) Tow, thread, weave
3. Handleability

C. ELECTROCHEMICAL

1. Platability
   - (a) Need for surface treatment
   - (b) Need for coating
2. Electrical Conductivity
3. Stability in plating bath

D. OTHER

1. Chemical Resistance (corrosion, oxidation, diffusion, etc.)
2. Mechanical Resistance (erosion, abrasion).

T A B L E 4.

FIBER CANDIDATES FOR ELECTRO-CO-DEPOSITION STUDIES

FIBER TYPE	DENSITY		DIAMETER		STRENGTH		MODULUS		S/D		E/D		COST $/lb.	AVAILABILITY
Vapor Deposited	Kg/m^3	$lb/in.^3$	mm	in.	MN/m^2	psi	GN/m^2	$x10^6$psi	$x10^5$ m	$x10^6$in.	$x10^6$m	$x10^7$in.		
B/W	$2.63x10^3$	0.095	0.10	0.004	311	450	400	58	1.22	4.8	15.7	62	250-500	Commercial (1000's lb/yr.)
B/SiO_2	$2.35x10^3$	0.085	0.10	0.004	228	330	380	55	0.99	3.9	14.7	58	--	Experimental
SiC/W	$3.46x10^3$	0.125	0.10	0.004	207	300	449	65	0.61	2.4	13.2	52	2100-3000	Pilot Plant < 100 lb/yr.
Borsic	$2.66x10^3$	0.096	0.10	0.0041	276	400	380	55	0.97	3.8	14.5	57	600-820	Commercial < 100 lb/yr.
$B_4C/B/W$	$2.35x10^3$	0.085	0.10	0.0041	207	300	428	62	1.17	4.6	18.5	73	--	Experimental
Glass														
(E)	$2.55x10^3$	0.092	0.010	0.0004	345	500	725	10.5	1.37	5.4	2.9	11.4	0.50	Commercial
(S)	$2.49x10^3$	0.090	0.010	0.0004	449	650	870	12.6	1.83	7.2	3.6	14.0	1.00	Commercial
(4H-1)	$2.66x10^3$	0.096	0.010	0.0004	504	730	100	14.5	1.93	7.6	3.9	15.4	--	Experimental
Graphite														
Thornel	$1.58x10^3$	0.057	0.0076	0.0003	173	250	276	40	1.12	4.4	17.8	70	550	Commercial (1000's lb/yr.)
RAE-60	$1.94x10^3$	0.07	0.0076	0.0003	207	300	414	60	1.09	4.3	21.8	86	200	Commercial (1000's lb/yr.)
RAE-HS	$1.85x10^3$	0.067	0.0076	0.0003	259	375	276	40	1.42	5.6	15.2	60	180	Commercial (1000's lb/yr.)
Whiskers														
Al_2O_3	$3.96x10^3$	0.143	0.0025 to 0.0127	0.0001 to 0.0005	1036	1500	428	62	2.72	10.7	11.2	44	12,000	Comm.40 lb/yr.
SiC	$3.21x10^3$	0.116	0.0025 to 0.0125	0.0001 to 0.0005	690	1000	497	72	2.21	8.7	15.2	60	250-13,500	Comm.20 lb/yr.

T A B L E 5.

CRITERIA FOR FIBER SELECTION

FIBER	DENSITY	STRENGTH	MODULUS	STRENGTH/WEIGHT	MODULUS/WEIGHT	DIAMETER	COST	AVAILABILITY
B/W	G	G	G	G	G	U	F	G
B/SiO_2	G	G	G	G	G	F-U	F	F
SiC/W	F	G	G	G	G	U	F	F
Borsic	G	G	G	G	G	U	F	F
$B_4C/B/W$	G	G	G	G	G	U	F	F
E-Glass	G	G	F-U	G	F	G	E	E
S-Glass	G	G	F-U	E	F	G	E	E
4H-1-Glass	G	G	F-U	E	F	G	F	F
TH-40 (graphite)	E	F	G	G	G	G	G	G
RAE-60	E	E	G	G	E	G	G	G
RAE-H.S.	E	E	G	G	G	G	G	G
Al_2O_3 (whiskers)	F	E	G	E	G	G	F	F
SiC (whiskers)	F	E	G	E	G	G	F	F

E=excellent
G=good
F=fair
U=unacceptable

TABLE 6.

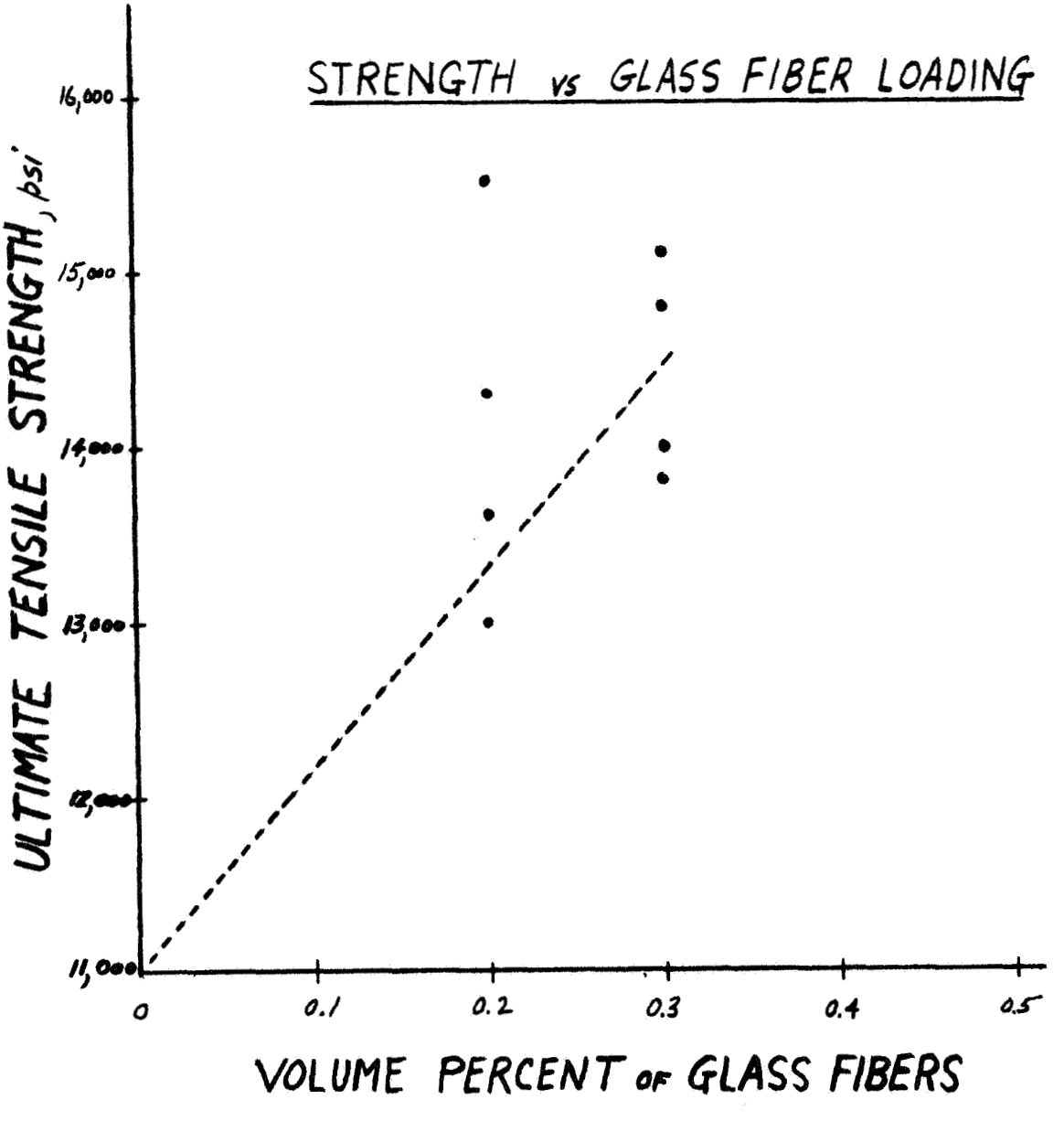
ULTIMATE TENSILE STRENGTH AND MODULUS OF ELASTICITY DATA FOR PLAIN(1), GLASS FIBER(1) AND FOR GRAPHITE FIBER INCORPORATED ELECTROFORMED ALUMINUM(1)

Specimen	U.T.S. Ultimate Tensile Strength		Elastic Modulus	
	GN/m^2	$x10^6$ psi	MN/m^2	ksi
1.Plain Electrofomed Aluminum	48.3	7.0	92.5	13.4
	47.4	6.87	102.	14.8
II. Glass Fiber-Aluminum Codeposit	49.2	7.13	94.6	13.7
	61.5	8.9	110.	15.9
	55.7	8.07	103	14.9
III. Graphite Fiber-Aluminum Codeposit				
A. Specimen, axis parallel to anode (longitudinal)	64.9	9.4	184	26.7
	64.9	9.4	171	24.8
B. Specimen, axis perpendicular to anode (transverse)	63.0	9.12	170	24.6
	62.6	9.06	159	23.0

(1) Test specimens taken from adjacent location to the three contract deliverable panels supplied to Langley Research Center.

FIGURE 1
PILOT CELL VOLUME FRACTION RUN DATA

Specimen Number	ULTIMATE TENSILE STRENGTH, psi	
	Pilot Cell Run #1	Pilot Cell Run #2
1.	13,000	13,800
2.	13,600	14,000
3.	14,300	14,800
4.	15,500	15,100



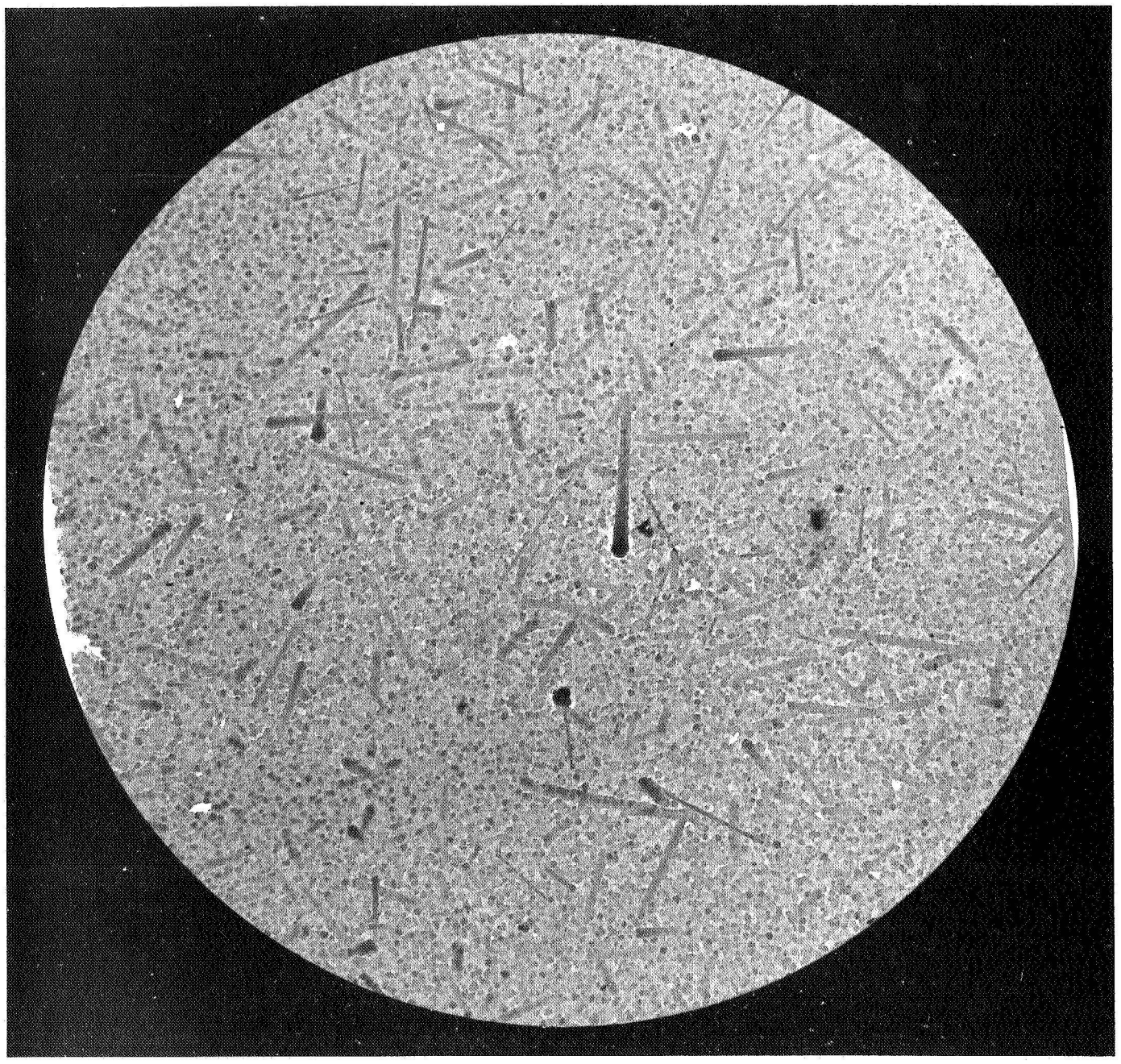
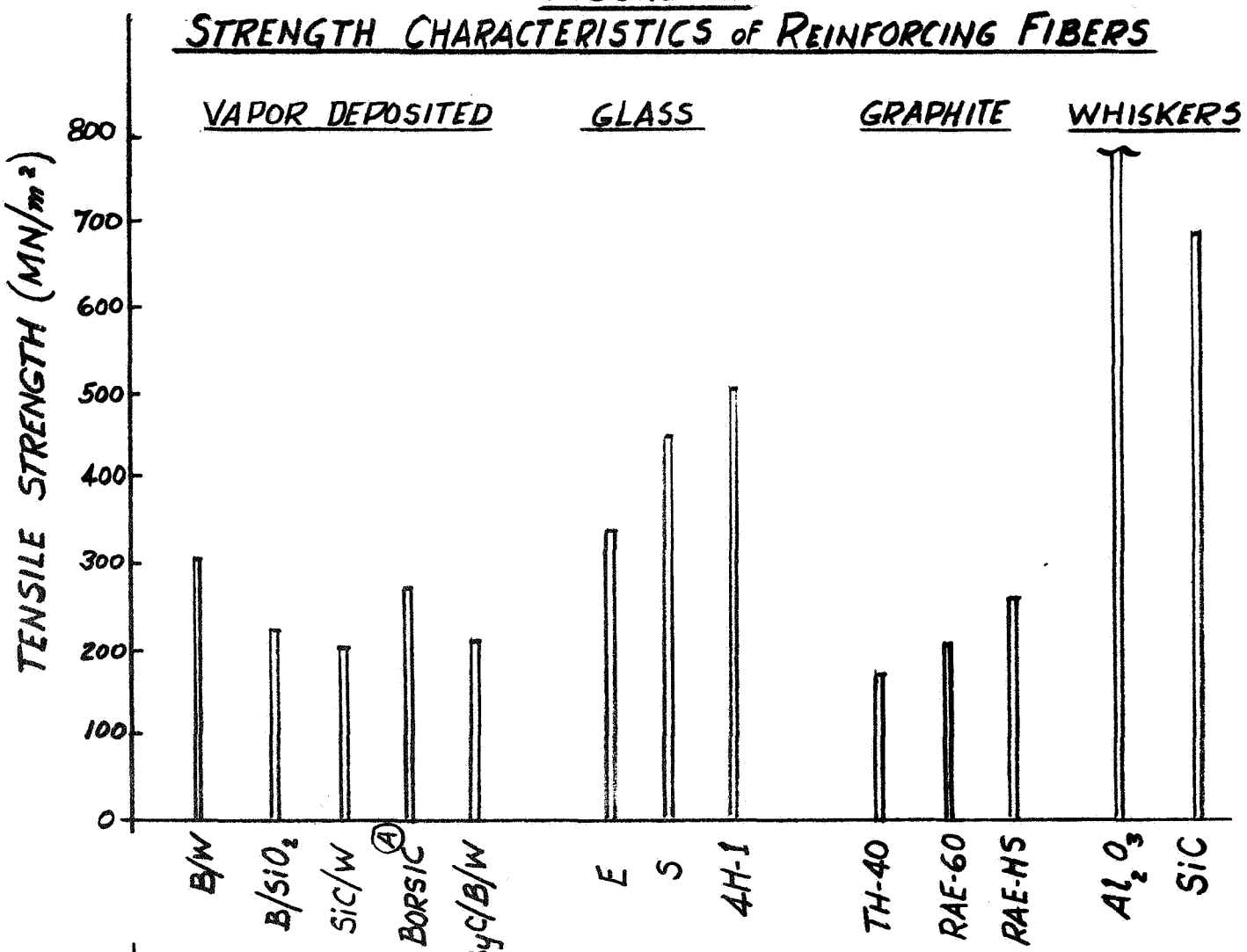


Figure 2. An x-ray microradiograph (17xmagnification) of the second pilot cell glass fiber-aluminum codeposition run, showing fiber distribution through the matrix. The near identical atomic number of aluminum and silicon makes contrast difficult. This problem would not exist for the graphite fiber-aluminum composites photographed by this non-destructive method.

FIGURE 3A

STRENGTH CHARACTERISTICS of REINFORCING FIBERS



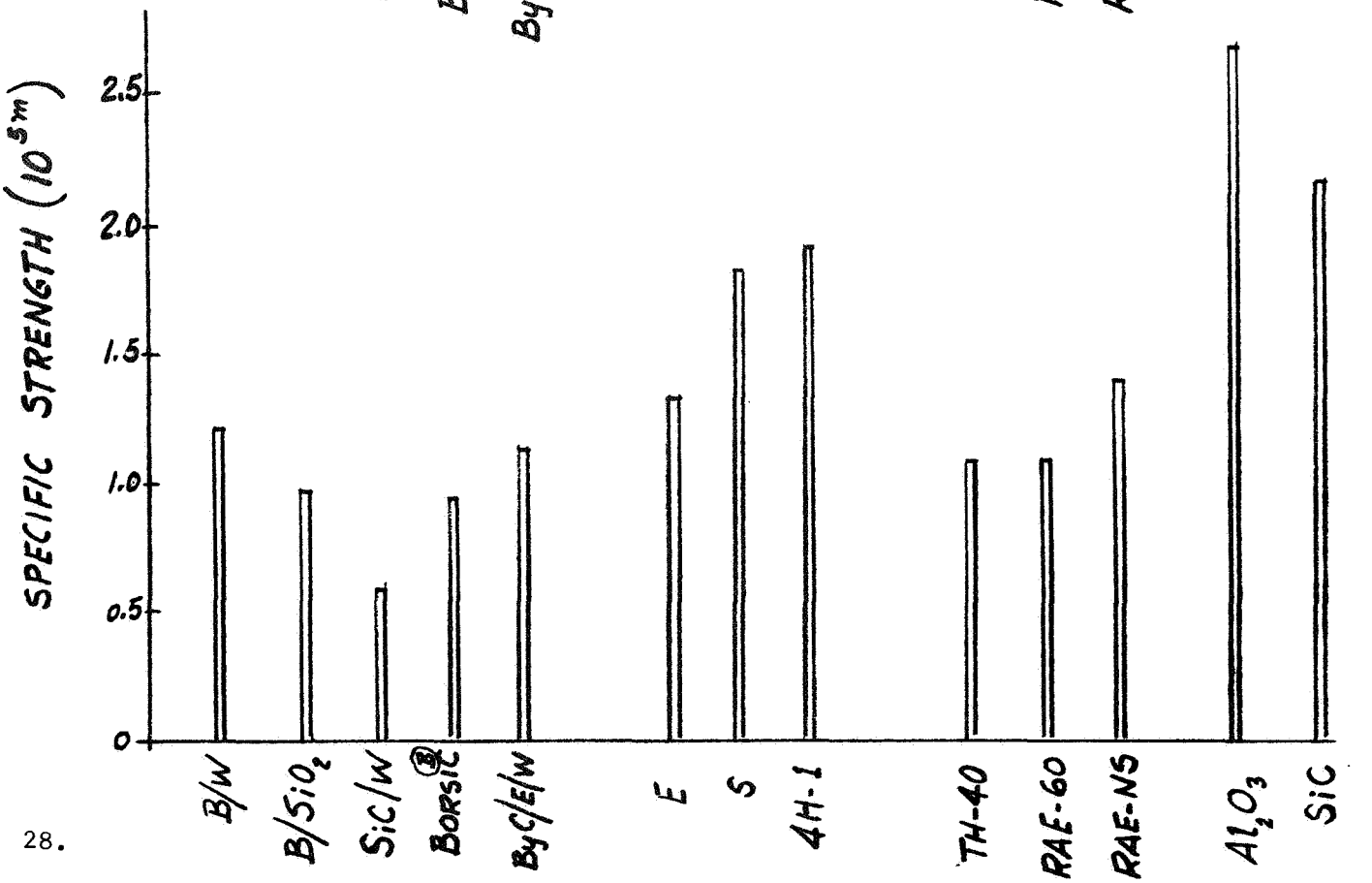
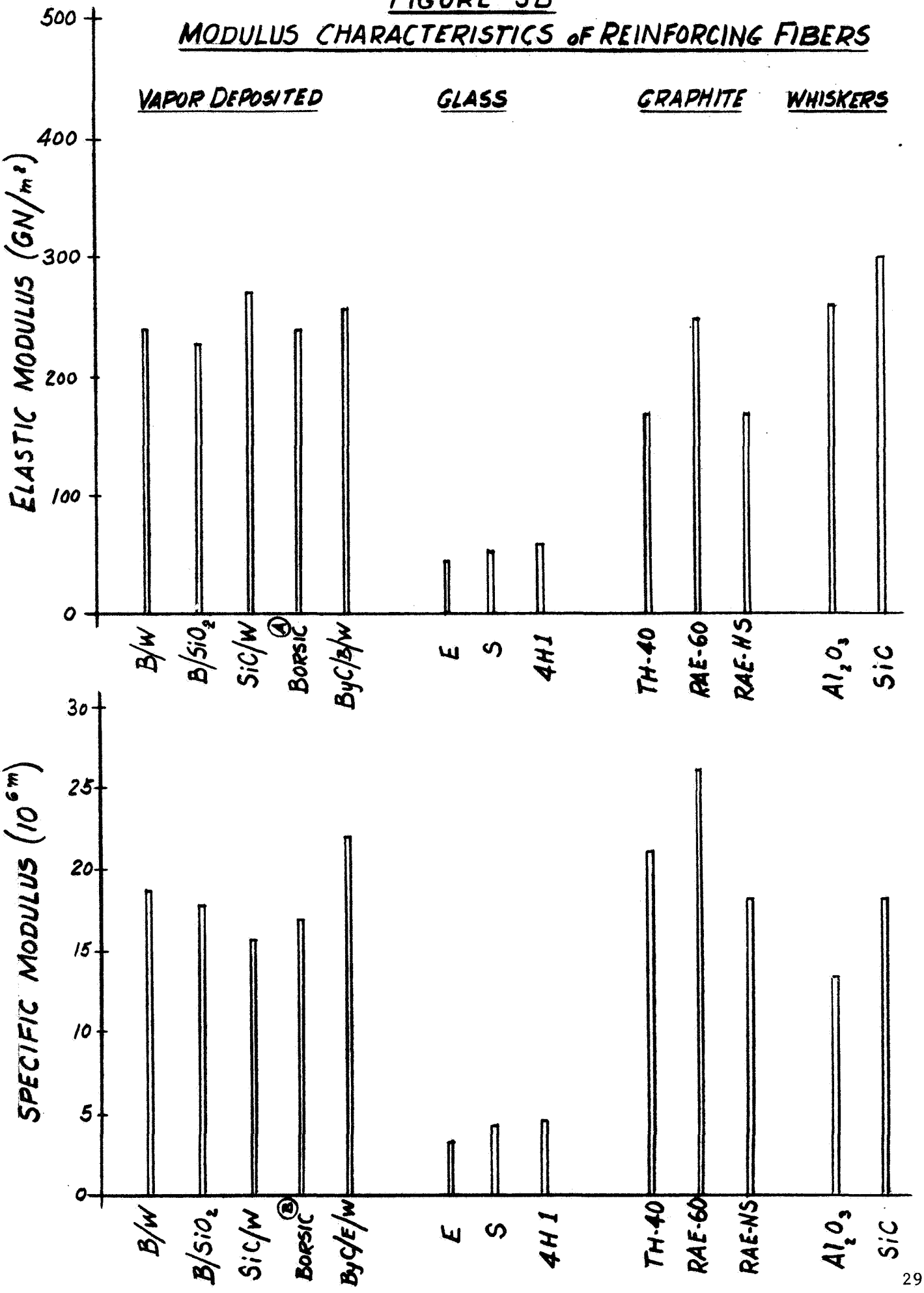
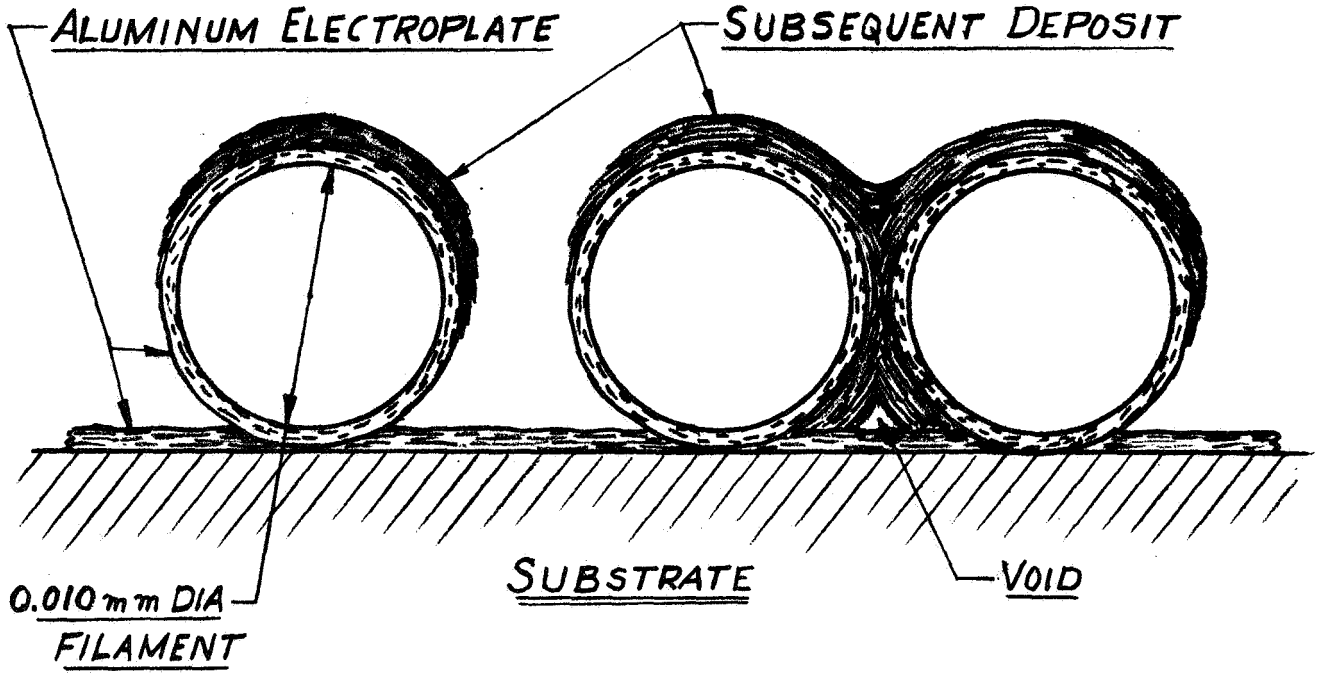


FIGURE 3B
MODULUS CHARACTERISTICS OF REINFORCING FIBERS





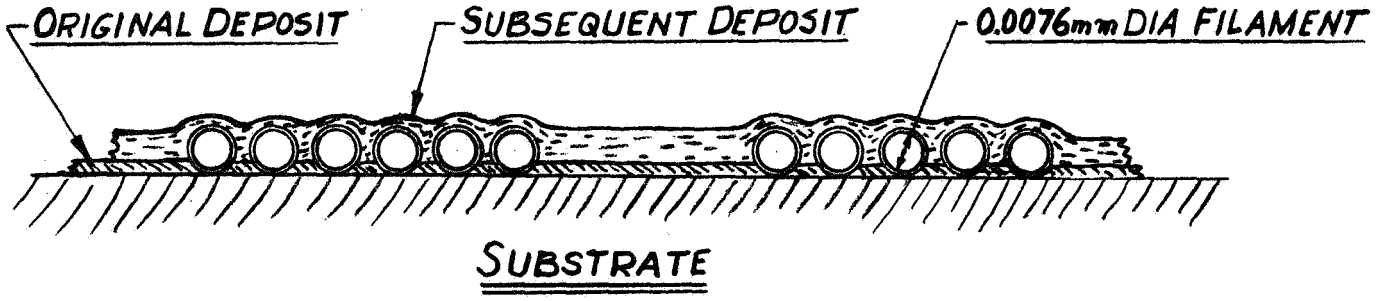


FIGURE 4

EFFECT OF FILAMENT DIAMETER ON MICROSTRUCTURE AND GEOMETRY OF ELECTROPLATED ALUMINUM

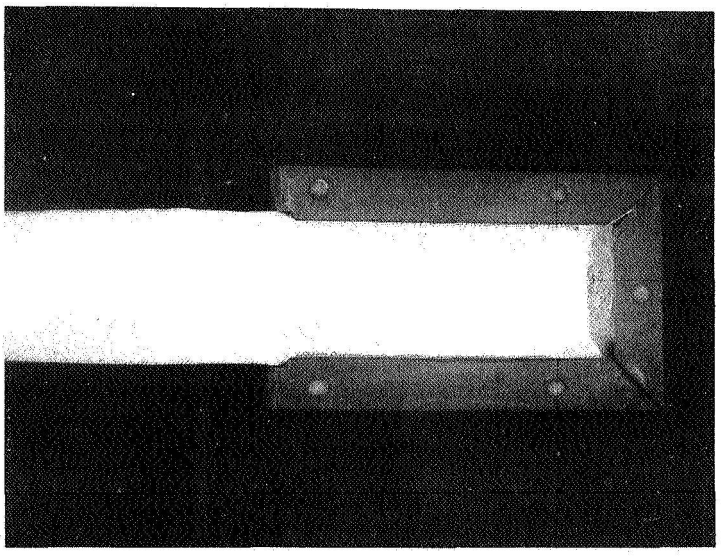


Figure 5. Glass cloth used to retain a fiber lay-up during initial plating.

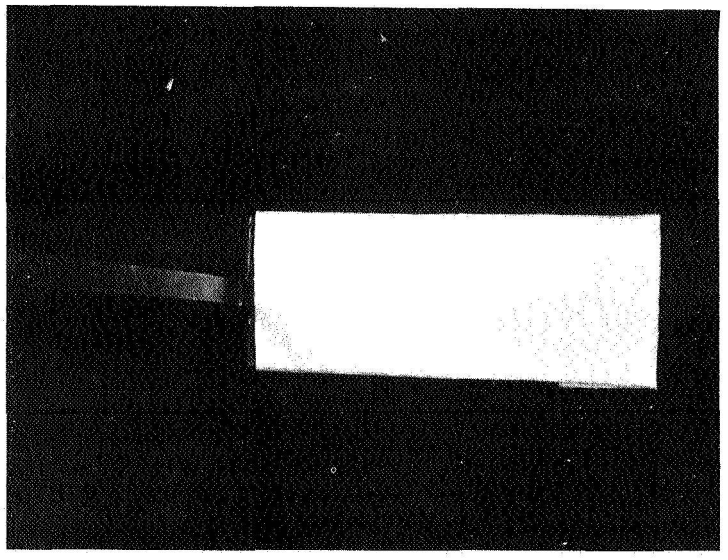


Figure 6. Glass filter "paper" used to retain a fiber lay-up during initial plating.

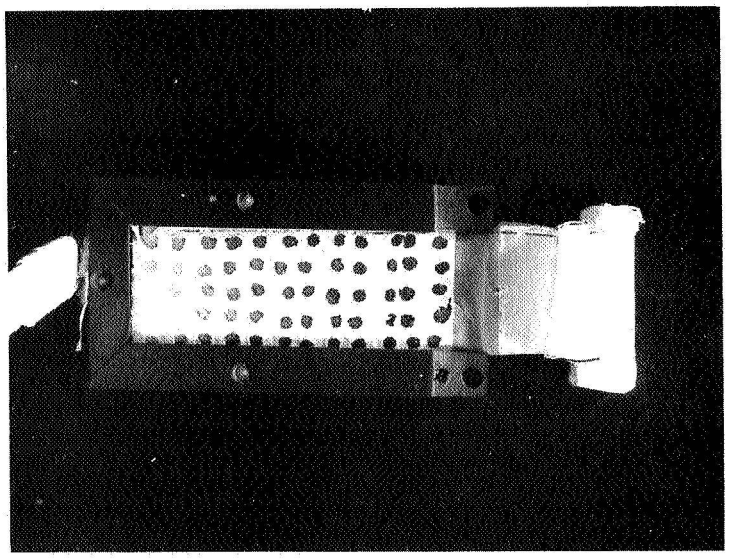


Figure 7. Perforated polytetrafluoroethylene film used to retain a fiber lay-up during initial plating.

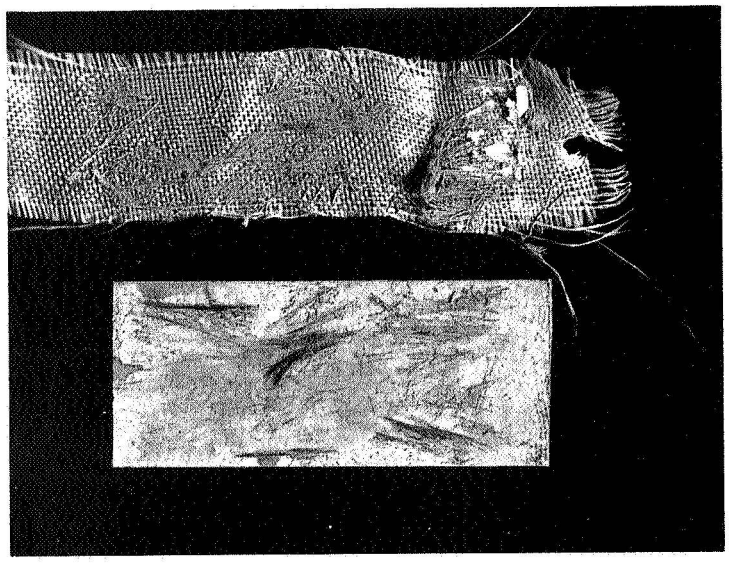


Figure 8. Glass cloth used to retain a fiber lay-up for Deposition Run 212-63 was removed. Note fibers "locked" into cloth by aluminum deposit.

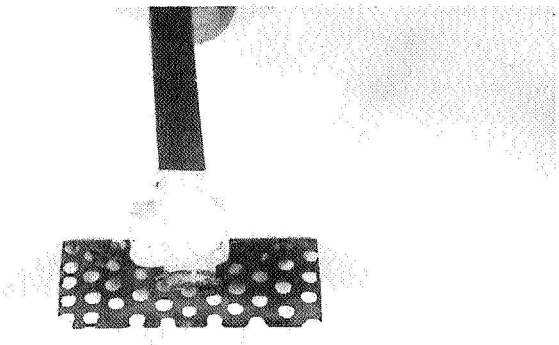


Figure 9. This perforated unplasticized PVC functioned best to retain a fiber lay-up during initial plating.

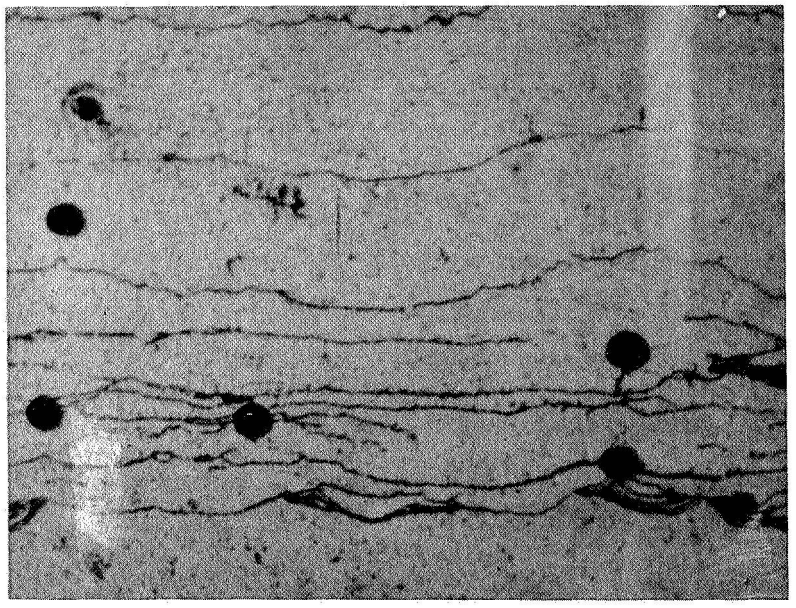


Figure 10. Photomicrograph of a transverse section of a graphite fiber-aluminum codeposited electroform which was etched with 1/2% HF for 30 seconds, reveals an intricate pattern of fine cracks in the micro-structure. Magnification is 530 times.

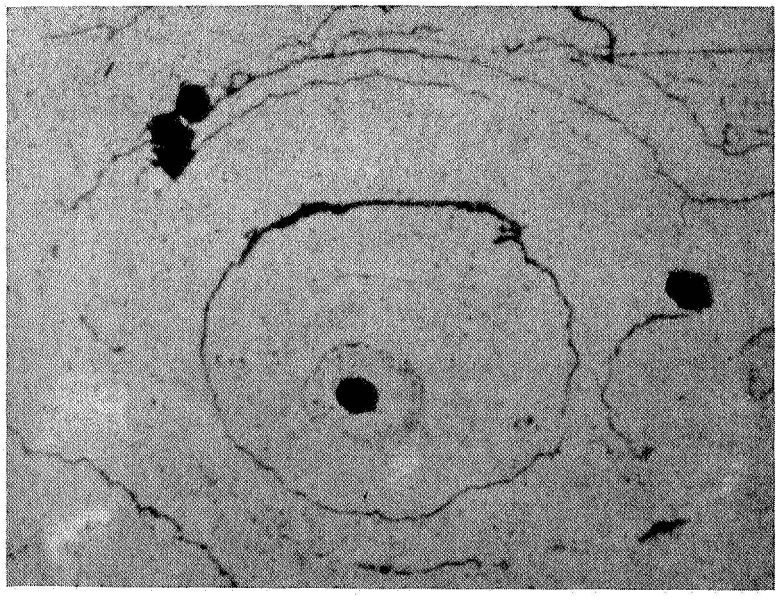


Figure 11. Photomicrograph of a longitudinal section of a graphite fiber-aluminum codeposited electroform which was etched with 1/2% HF for 30 seconds, indicates a different pattern of fine cracks in the micro-structure, as compared with Figure 2. Magnification is 530 times.

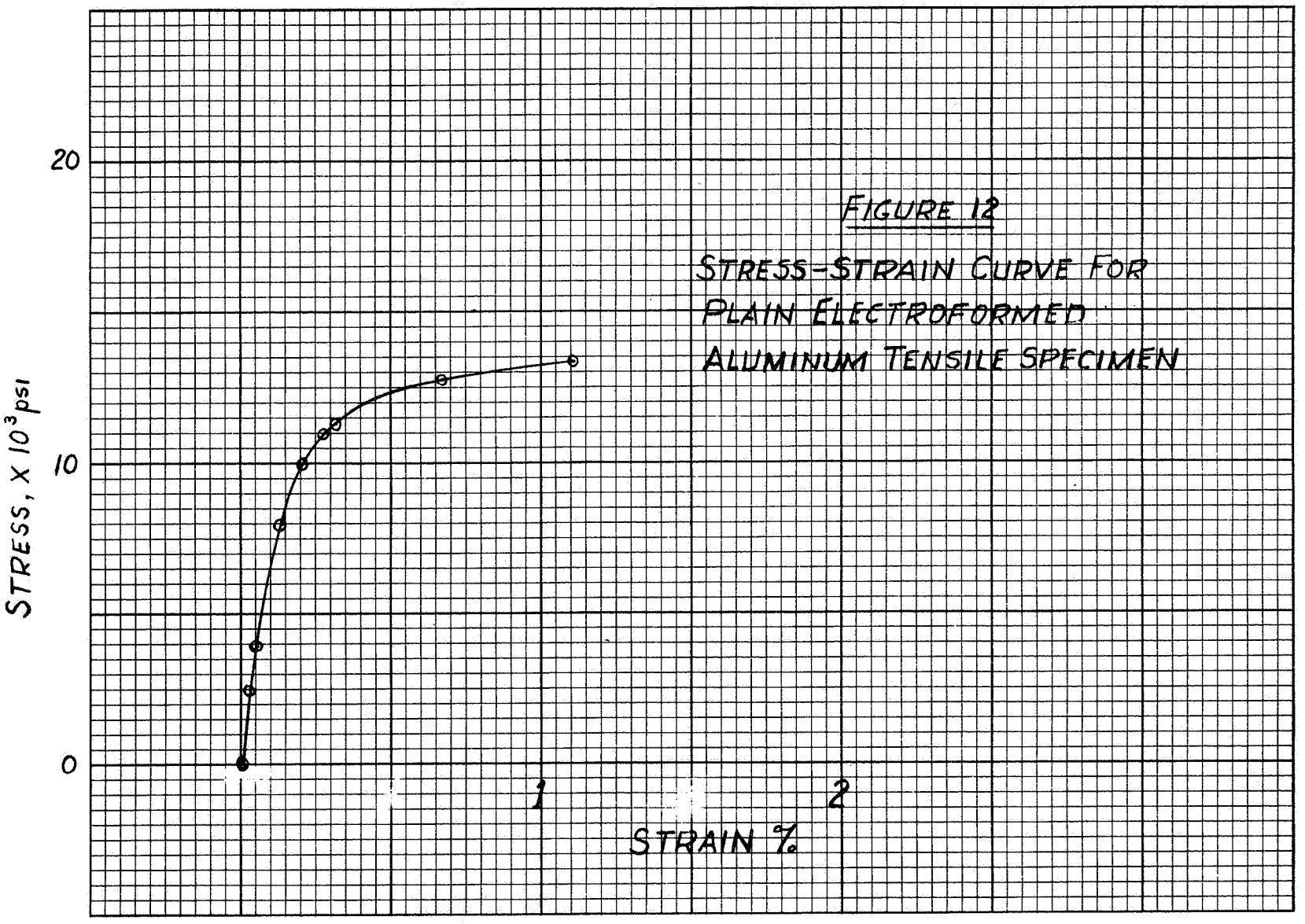


FIGURE 12

STRESS-STRAIN CURVE FOR PLAIN ELECTROFORMED ALUMINUM TENSILE SPECIMEN

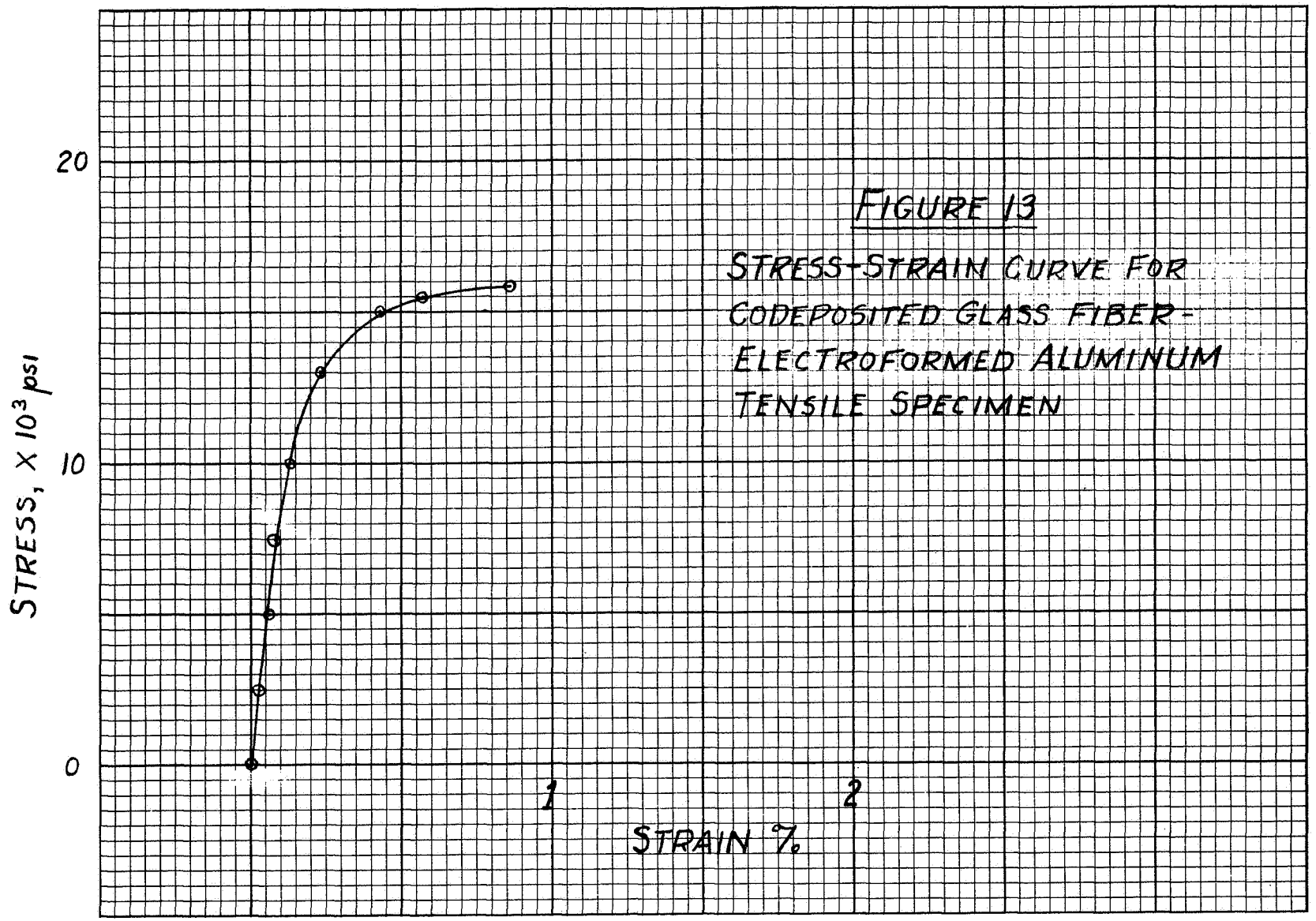


FIGURE 13

STRESS-STRAIN CURVE FOR CODEPOSITED GLASS FIBER-ELECTROFORMED ALUMINUM TENSILE SPECIMEN

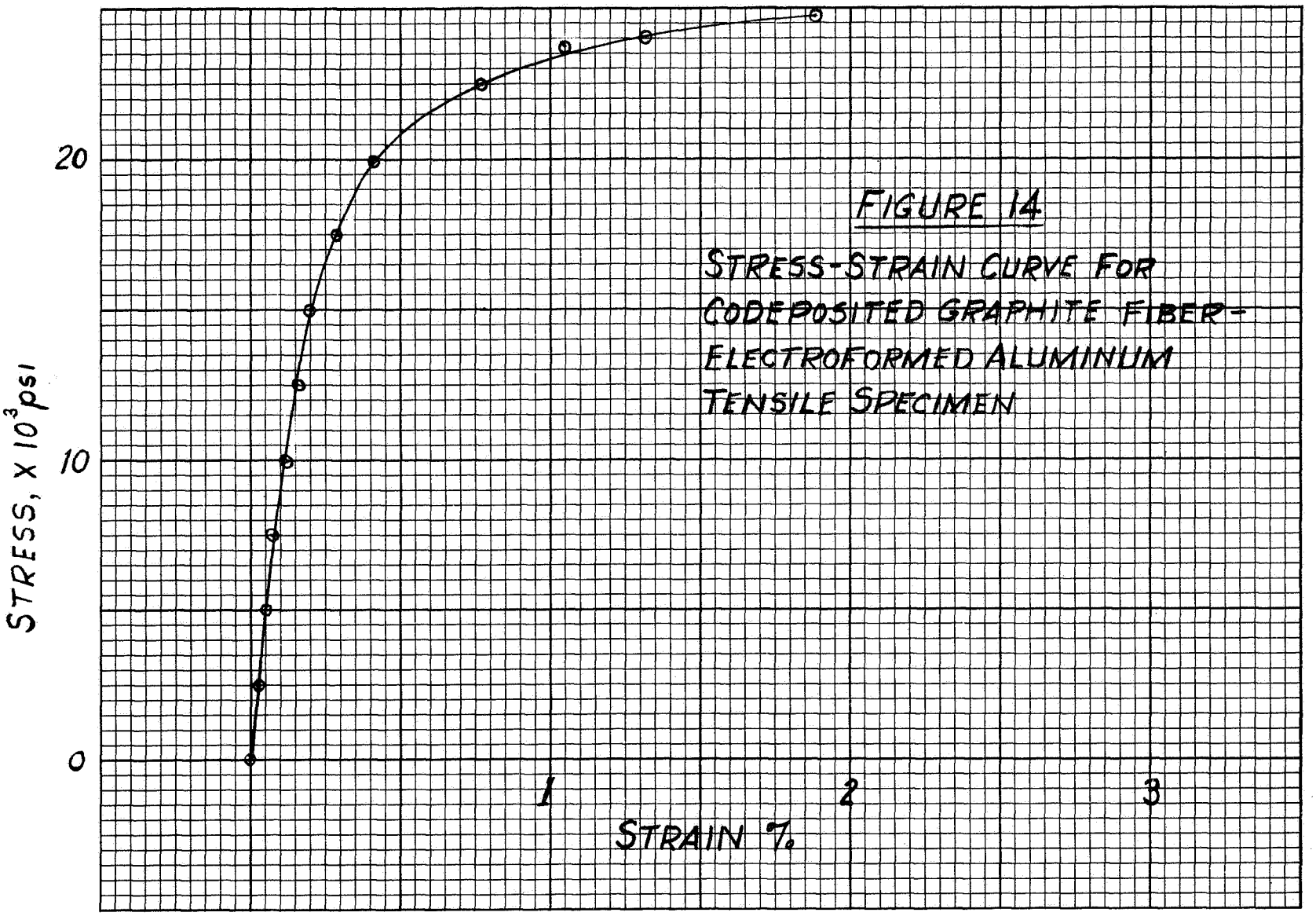


FIGURE 14

STRESS-STRAIN CURVE FOR CODEPOSITED GRAPHITE FIBER-ELECTROFORMED ALUMINUM TENSILE SPECIMEN

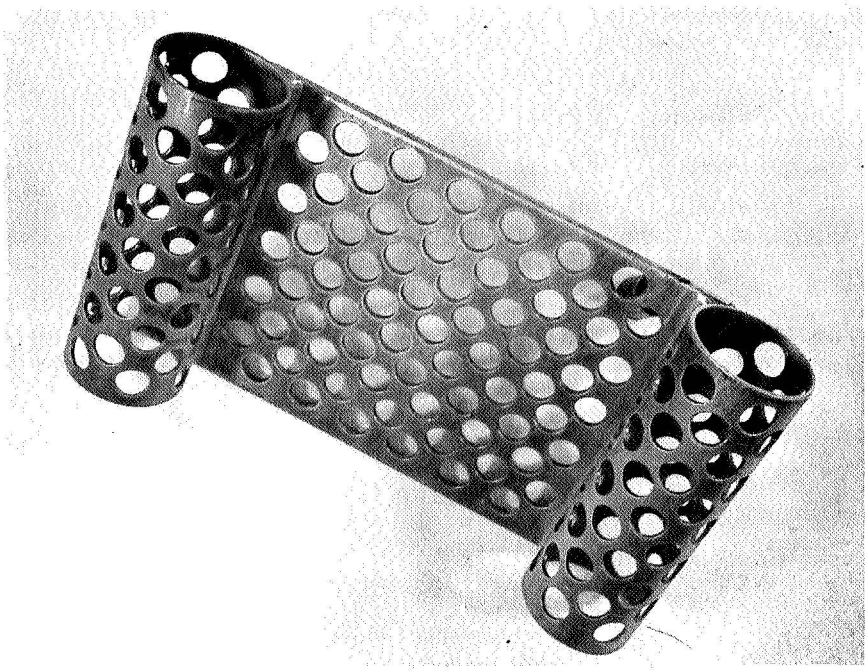


Figure 15. This prototype copper mandrel for electroforming a structural element with upper tubular support, was used to establish the current profile for this specific geometry.

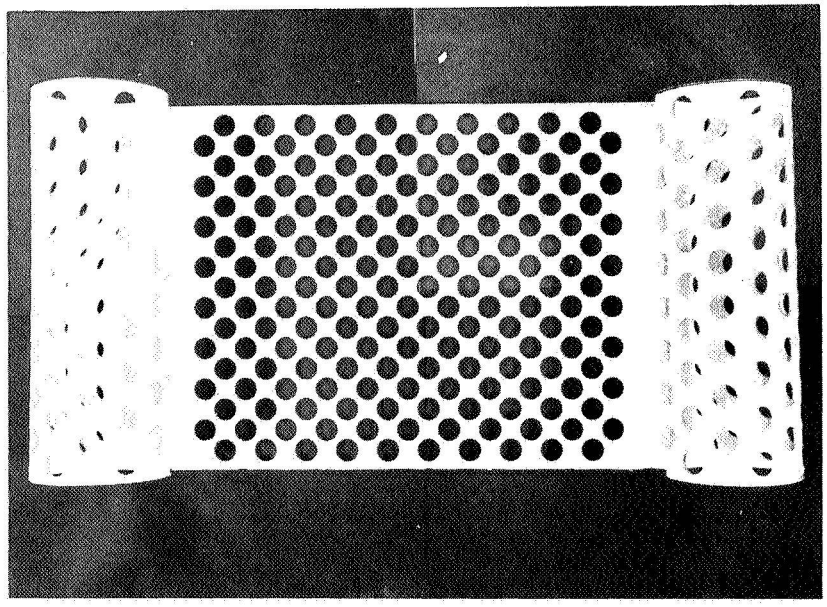


Figure 16. The graphite fiber-aluminum structural element was electroformed in the pilot cell to demonstrate the complexity of shape which could be fabricated.

APPENDIX A

The following is taken from Dr. Musikant's study on the "Streaming Solution" process.

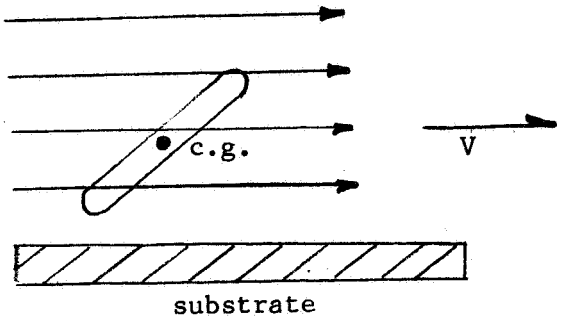
1. Turbulance in the stream will defeat efforts to align the fibers. The maximum velocity for laminar flow of a fluid with the approximate viscosity and density of the electrolyte is given below for full round tubes:

Tube Diameter	Max. Velocity Laminar Flow
25.4 mm (1 in.)	16.5 mm/sec. (0.65 in./sec.)
12.7 mm (1/2 in.)	33.0 mm/sec. (1.3 in./sec.)
6.4 mm (1/4 in.)	66.0 mm/sec. (2.6 in./sec.)

Thus, very low velocities and small diameter tubes are required for laminar flow. Similarily, for open channel flow, a low velocity is required for laminar flow.

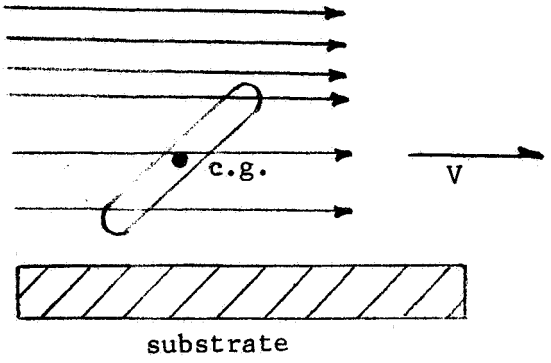
2. Each individual fiber has its c.g. at the center of pressure (simple cylinder) in a uniform flow field. If the axis of a fiber is misaligned with the flow direction in a uniform velocity field, there is no tendency to rotate the fiber. In a laminar flow field, near the wall a restoring moment will be developed to align the fiber with the flow direction because of the velocity gradient.

   For open channel flow, a sharp velocity gradient exists for the lower 2.54 mm (0.1 inch). Fibers in this region will be rotated due to the velocity gradient and will tend to tumble downstream because there is no stable orientation. Neither will the flow field have any tendency to align fibers with the channel centerline, if they are initially skewed to the direction offluid flow.



Uniform Flow Field

No restoring moment



Non-uniform Flow Field

Creates a restoring moment but no stable position.

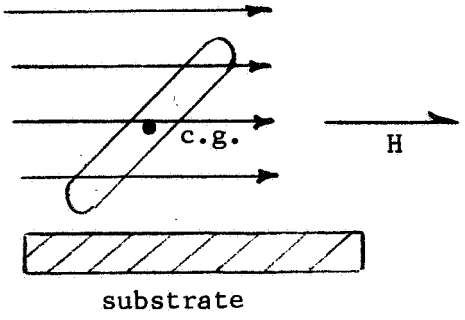
3. If the fibers are coated with ferromagnetic metal, then alignment possibilities exist by use of a magnetic field. However, if the ferromagnetic coating is unmagnetized prior to incorporation in the electrolyte bath, a uniform magnetic field will produce no restoring moment.

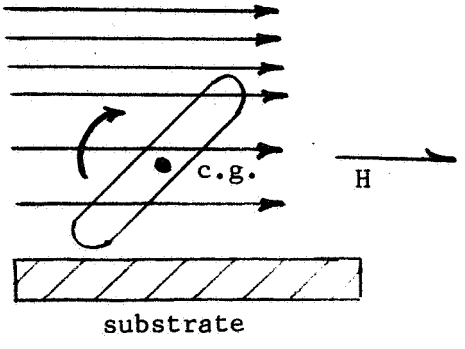
Uniform Magnetic Field
Unmagnetized Ni-Coated Fiber

No restoring moment

Non-Uniform Magnetic Field
Unmagnetized Ni-Coated Fiber

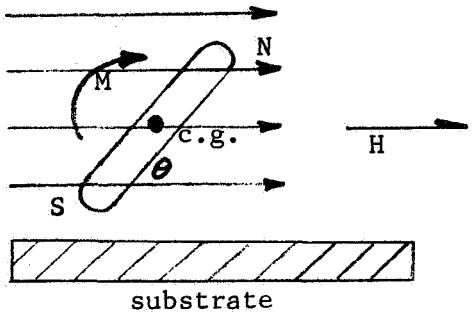
Creates a restoring moment





If a non-uniform magnetic field can be established, then a restoring moment will be developed. However, an extremely large and perhaps unattainable magnetic gradient must be established to effect rotation and alignment of unmagnetized fibers by this means.

4. As an example, a Ni-coated graphite fiber can be permanently magnetized by passing the continuous filament through a magnetic field of 94.5 A/m (12 oersteds). In this circumstance, the Ni will achieve an induction of about 0.4 tesla (4000 gauss). A fiber so magnetized and injected into a uniform magnetic field will experience a strong restoring moment, always tending to align the fiber with the direction of the uniform magnetic field.



Uniform Magnetic Field
Magnetized Ni-Coated Fiber

creates a restoring moment and a stable position aligned with field.

Calculations indicate that fibers with a Ni-coating of 1 μ or less in a 0.1 tesla (1000 gauss) or smaller field will be aligned very wuickly. The fluid will tend to damp oscillations.

5. Coatings which develop passive oxide films cannot accept an adherent Al electrodeposited film without prior surface activation. Many ferromagnetic materials are available composed of Fe, Ni, and/or Co alloyed with other non-magnetic metals. Thus the problem of suitable activation can be solved by proper choice of alloy and/or chemical treatment of the coated fibers prior to immersion in the electrolyte.

Based on these considerations (which can be refined), a process, such as depicted in Figure A-1, appears feasible. Graphite fibers, Ni-coated, magnetized, and cut to about 1 mm length are agitated in a reservoir of electrolyte. The mixture is metered through a metering valve and injected into an open rectangular channel at a velocity of about 66 mm/sec. (2.6 in/sec.). The channel depth is maintained at 6.4 mm (1/4-inch).

A solenoid, surrounding the channel, imposes a magnetic field on the fibers which aligns them. The fibers settle due to the influence of gravity and are captured by the Al electrodepositing on the Cu substrate cathode. The mixture of fibers and electrolyte is recirculated by means of a suitable pump.

The problem which may arise is the tendency for the fibers to clump together. This tendency may be controlled by use of a sufficiently dilute solution. A minimum degree of magnetization of the Ni-coating will be used to minimize the clumping effect. Parameters for investigation are:

(1) Ferromagnetic alloy composition which is compatible with the aluminum electrodeposition process.

(2) Fiber size, thickness of coating and magnetization level.

(3) Flow rate, channel depth, channel dimensions, length of channel needed for gravity settlement of fibers.

(4) Concentration ratio of fiber to electrolyte.

(5) External magnetic field strength.

(6) Anode position, configuration, and electrical parameters for most favorable aluminum deposition.

(7) Application of technique to other substrate configurations, such as a full cylinder.

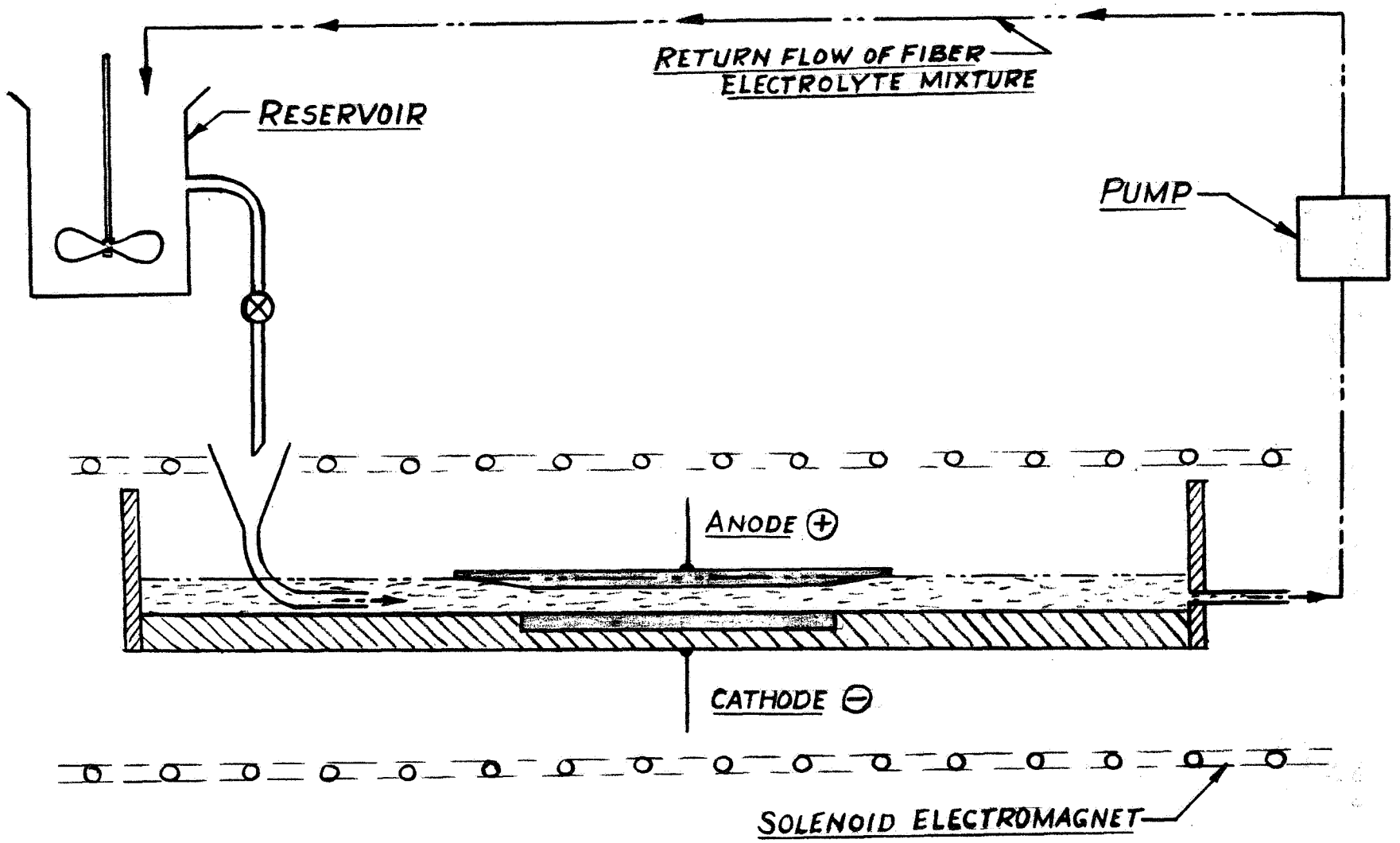


FIG.A1 :- PASSING MAGNETIZED FIBERS THROUGH A MAGNETIC FIELD

APPENDIX B

The following was taken from R.L. Mehan's internal report on the shear strength of the graphite fiber-electrodeposited aluminum bond:

It was deemed important to determine quantitatively the bond strength between carbon fibers and the aluminum matrix, in which they are imbedded as fabricated by the electrodeposition technique. The need for these data are readily evident. Because carbon and aluminum are known to be inert with respect to one another below approximately 1073°k (800°C)[1], there seemed little likelihood of a strong chemical bond between carbon fibers and aluminum when the system is formed by electroplating at or near room temperature. If a very weak bond were present, the critical transfer length, ℓ_c, would be inordinately large. The quantity, ℓ_c, is given by [2]:

$$\ell_c = \frac{\sigma_f}{2\tau} d \qquad (1)$$

where

σ_f = fiber fracture stress

d = fiber diameter

τ = matrix shear strength or interfacial bond shear strength, whichever is lower (assumed constant).

A large value of ℓ_c in turn would require that the actual fiber length would have to be large in order to achieve significant composite strength, given by[2]:

(1) R.E. Nightingale, "Various Forms of Carbon", in H.H. Hausner and M.C. Bowman, ed. Fundamentals of Refractory Compounds, Plenum Press, N.Y. 1968

(2) A. Kelly, C.J. Davies, Met. Review 10, 1 (1965)

$$\sigma_c = \sigma_f V_f \left(1 - \frac{\ell_c}{2\ell}\right) + \sigma_{m'} \left(1 - V_f\right) \qquad (2)$$

where

σ_c = composite strength

V_f = volume fraction fibers

ℓ = fiber length

$\sigma_{m'}$ = strength of matrix at the strain at which the fibers fail

If $\ell < \ell_c$, then the composite will always fail by the plastic strain of the matrix. Although some reinforcement will occur, the degree would not be large. For this case the composite strength is given by[2]:

$$\sigma_c = \sigma_\mu \left(1 - V_f\right) + \frac{\tau \ell}{d} V_f \qquad (3)$$

where

σ_μ = ultimate strength of the matrix

It is quite important, therefore, to have the critical transfer length greater than the fiber length used. As may be seen from equation (1), this depends inversely on the interfacial shear strength. It is not necessary, of course, to have a strong chemical bond between the fiber and matrix for effective reinforcement. In some cases, such as glass-epoxy composites, frictional forces are sufficient.

For the fibers used in the NASA program and in this brief investigation, the manufacturer claims the following properties:

Density: 1.95 gms/cc
Tensile Strength: 250-300 ksi
Elastic Modulus: 50-60 x 10^6 psi

Initially in this investigation, the base-line fiber properties were determined. The fibers themselves are shown in Figure B1. From a photomicrograph similar to this, an average fiber diameter of 7.1×10^{-3} mm (2.8×10^{-4} in.) was determined (average of 26 random observations).

The tensile tests were conducted in a micro-tensile testing machine, using techniques previously described.[3] The breaking load and extension were recorded, and the strength and modulus computed from the average area. Only for specimen #1 was the actual fiber area measured after fracture.

The data are shown in Table B1. As can be seen, excellent agreement with the manufacturer's specification were obtained. The only exception, specimen #3, had a lower than average modulus. This is attributed to a larger than average carbon fiber. Good agreement was also obtained between the individual fiber diameter actually measured and the assumed values. For the measuring technique used, see Reference 3.

The technique used to determine the interfacial shear strength involved the use of direct pull-out tests. The principle is illustrated in Figure B2. If the fiber is imbedded in the aluminum as shown in Figure B2A, the fiber will break. As the distance, ℓ, becomes shorter, a value will eventually be reached when the fiber will pull out. A further decrease in imbedded length will cause a further reduction in the load necessary to pull the fiber away from the aluminum. This interfacial shear stress can be approximately computed by equating the force in the fiber to the resistance in the aluminum by shear. If the load in the fiber is P, then for pull-out to occur we have:

$$P = \tau \pi d \ell \qquad (4)$$

The load P is more conveniently expressed in terms of the fiber stress

$$\sigma \frac{\pi}{4} d^2 = \tau \pi d \ell \qquad (5)$$

$$\text{OR} \quad \sigma = \frac{4\tau}{d} \ell$$

Equation (5) states that a plot of σ vs. ℓ should yield a straight line of slope $\frac{4\tau}{d}$, assuming the shear strength, τ, is a constant.

The shear strength, however, is not a constant, as may be seen from examining Figure B2. The quantity actually determined is an "average" shear strength, useful for order of magnitude measurements, but not to be construed as an accurate figure.

The carbon fibers were individually mounted on a rack, as shown in Figure B3. They were glued to the rack using a silver base paint to allow current flow. One-half of the fiber lengths were electroplated with about 0.085 mm (.003 in.) thick aluminum. Individual test specimens were then prepared, formed by cutting-off the aluminum portion to the desired length. The specimen was then mounted in the micro-tensile testing machine, as shown in Figure B4. Considerable care was exercised to avoid having the cement (diphenylcarbazide) come into contact with the fiber where it was embedded into the aluminum. Any specimen showing signs of cement-fiber contact near the aluminum was rejected. A photograph showing a particular specimen glued onto an anvil before insertion into the testing machine is shown in Figure B5.

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(3) R.L. Mehan, E. Feingold, J. Materials, 2, 239 (1967).

The results obtained are shown in Figure B6. The slope of the initial line, from equation (5), is given by:

$$\frac{\sigma}{\ell} = 5.98 \times 10^{11} \frac{kg}{m^3} = \frac{4\tau}{d}$$

Using the average value of d of 7.1×10^{3} mm (2.8×10^{-4} in.), the value of τ is found to be

$$\tau = 10.4 \text{ MN/m (1510 psi)} \qquad (6)$$

It should be remembered this is an average value because of the non-linear variation of τ with respect to the interface length.

Using the value of τ given by equation (6), the critical length, ℓ_c, may be calculated from equation (1)

$$\ell_c = \frac{\sigma_f d}{2\tau} = \frac{2430}{(2)(10.4)} (7.1 \times 10^{-3}) \qquad (7)$$

$$\ell_c = 0.91 \text{ mm } (0.033 \text{ inch})$$

If the fiber length used to fabricate the electroformed composites is greater than 0.91 mm (0.033 inch), fiber reinforcement will occur (provided, of course, sufficient fibers are present to exceed the critical volume fraction necessary for reinforcement, previously found to be of the order of 1.5%).[4] Because the fiber lengths used for fabricating experimental aluminum-graphite composites were considerably longer than this, it may be concluded that the bond strength between the carbon fiber and the electroplated aluminum is not a limiting problem for obtaining composite reinforcement at room temperature.

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(4) R.L. Mehan, PIR 2410-343, October 2, 1968.

TABLE B.1 MODULUS AND STRENGTH OF TESTED CARBON FIBERS

Specimen	Gage Length (mm)	Elastic Modulus GN/m^2	Elastic Modulus psi	Ultimate Stress GN/m^2	Ultimate Stress ksi
1*	5.65	371	53.8×10^6	3.22	466
2**	8.96	417	60.4×10^6	2.89	416
3**	11.37	300	43.4×10^6	2.36	342
4**	9.59	366	53.0×10^6	2.01	291
5**	6.74	367	53.2×10^6	2.18	316
6**	7.57	403	58.4×10^6	2.18	316
7**	5.75	--	--	2.18	316
			Avg.	2.43	352

*Area measured - 3.77×10^{-5} mm^2 (5.85×10^{-8} in^2)

**Area measured - 4.19×10^{-5} mm^2 (6.5×10^{-8} in^2)

*** Area probably too large

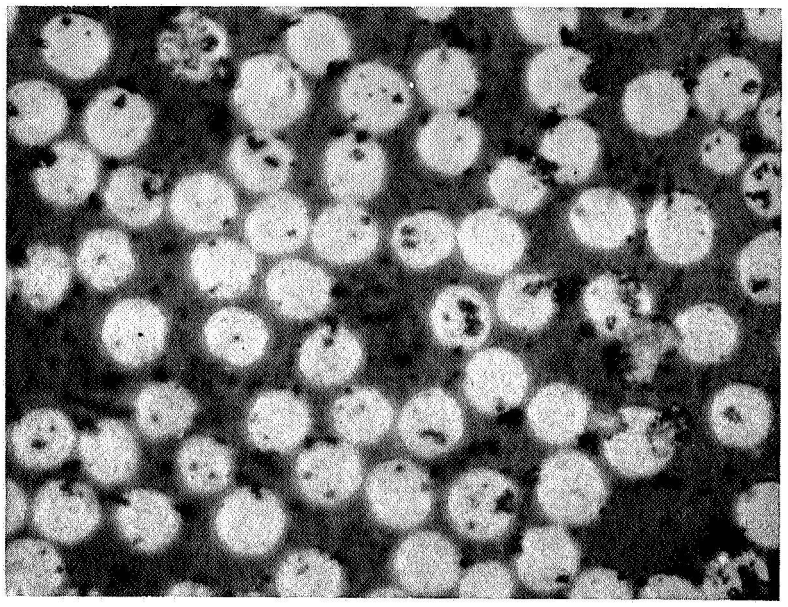
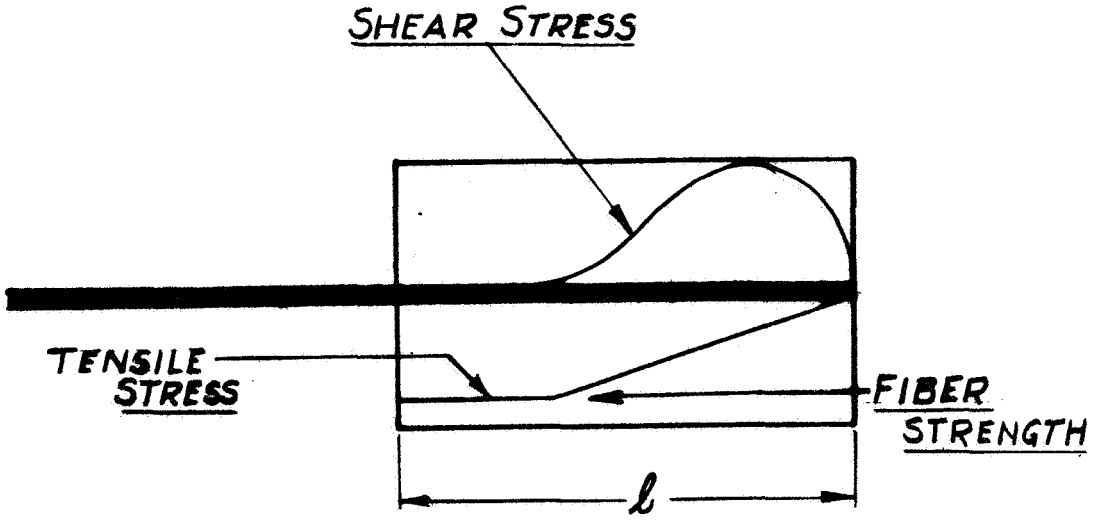
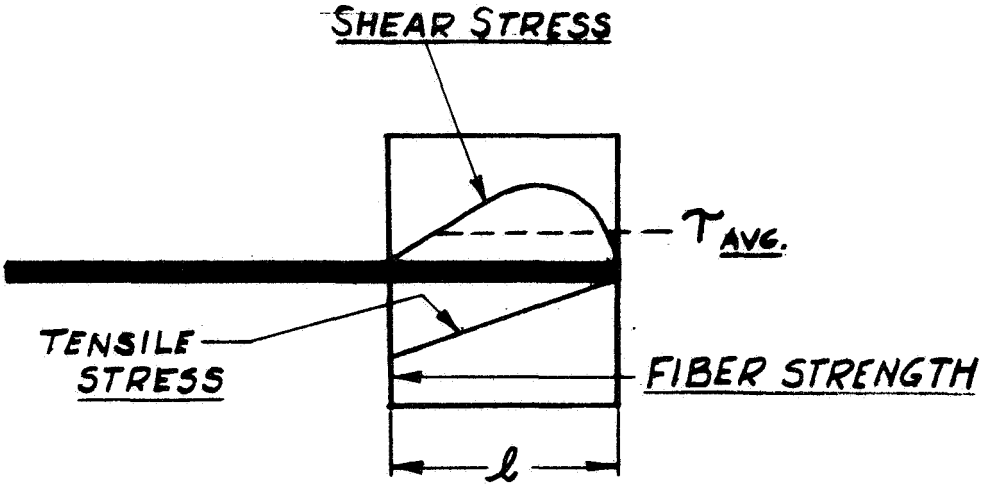


Figure B1. Photomicrograph of Courtauld Type B Carbon Fibers
Magnification - 1100 X



A-FIBER FAILURE



B- FIBER PULL-OUT

FIG. B2 :- SCHEMATIC REPRESENTATION OF INTERFACIAL SHEAR STRESSES

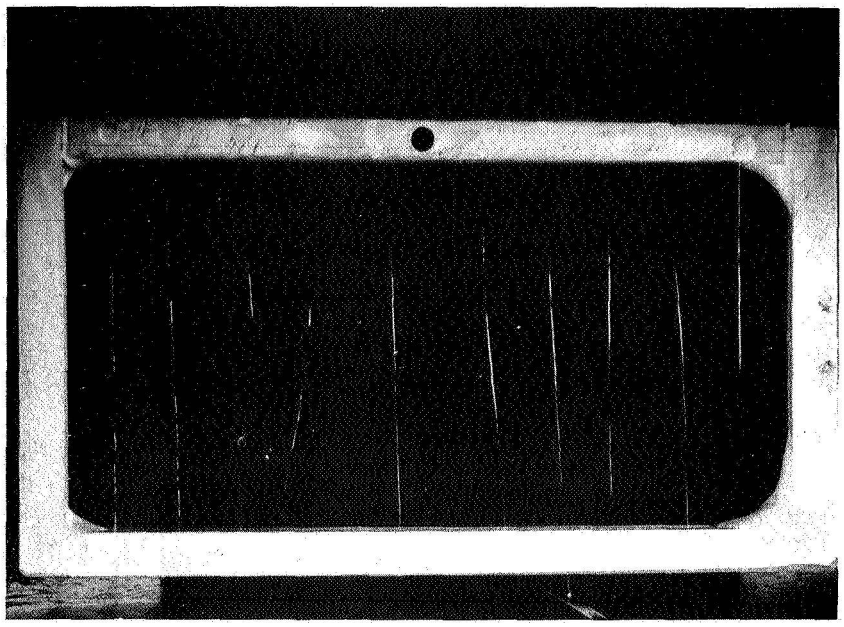


Figure B3. Photograph of rack with fibers glued in place.
Magnification 2X

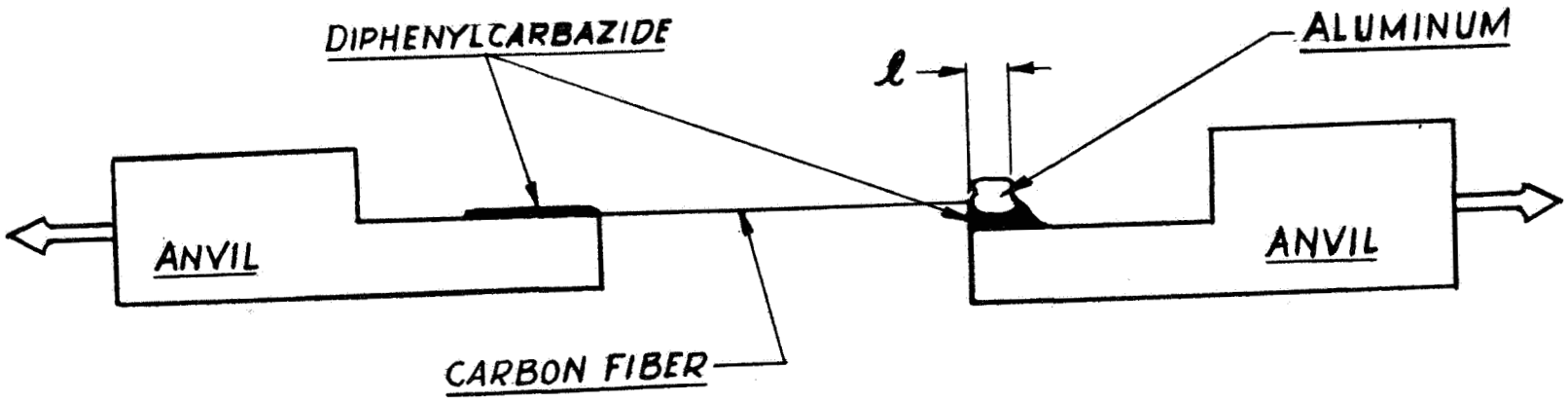


FIG.B4:- EXPERIMENTAL SET-UP FOR TESTING

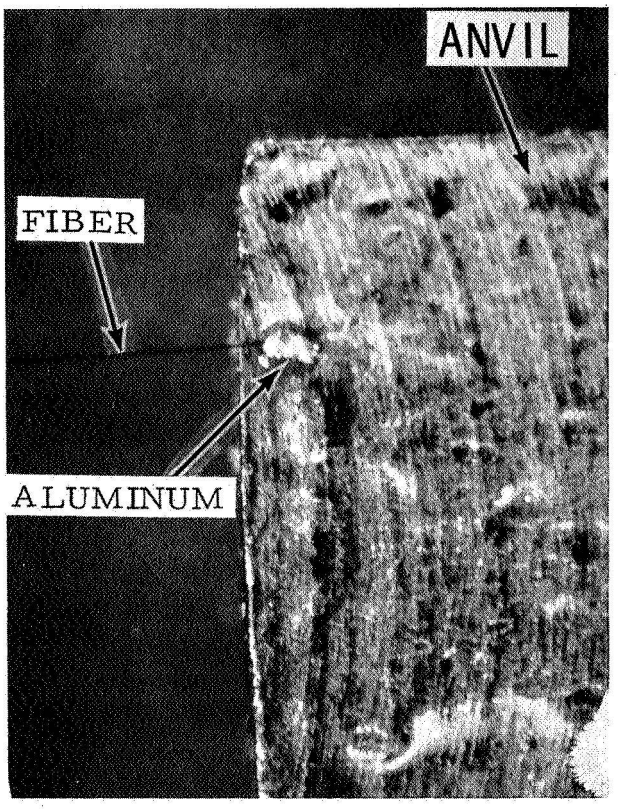


Figure B5. Photograph of tensile machine anvil with aluminum-carbon fiber specimen glued in place. Mag. 42 X

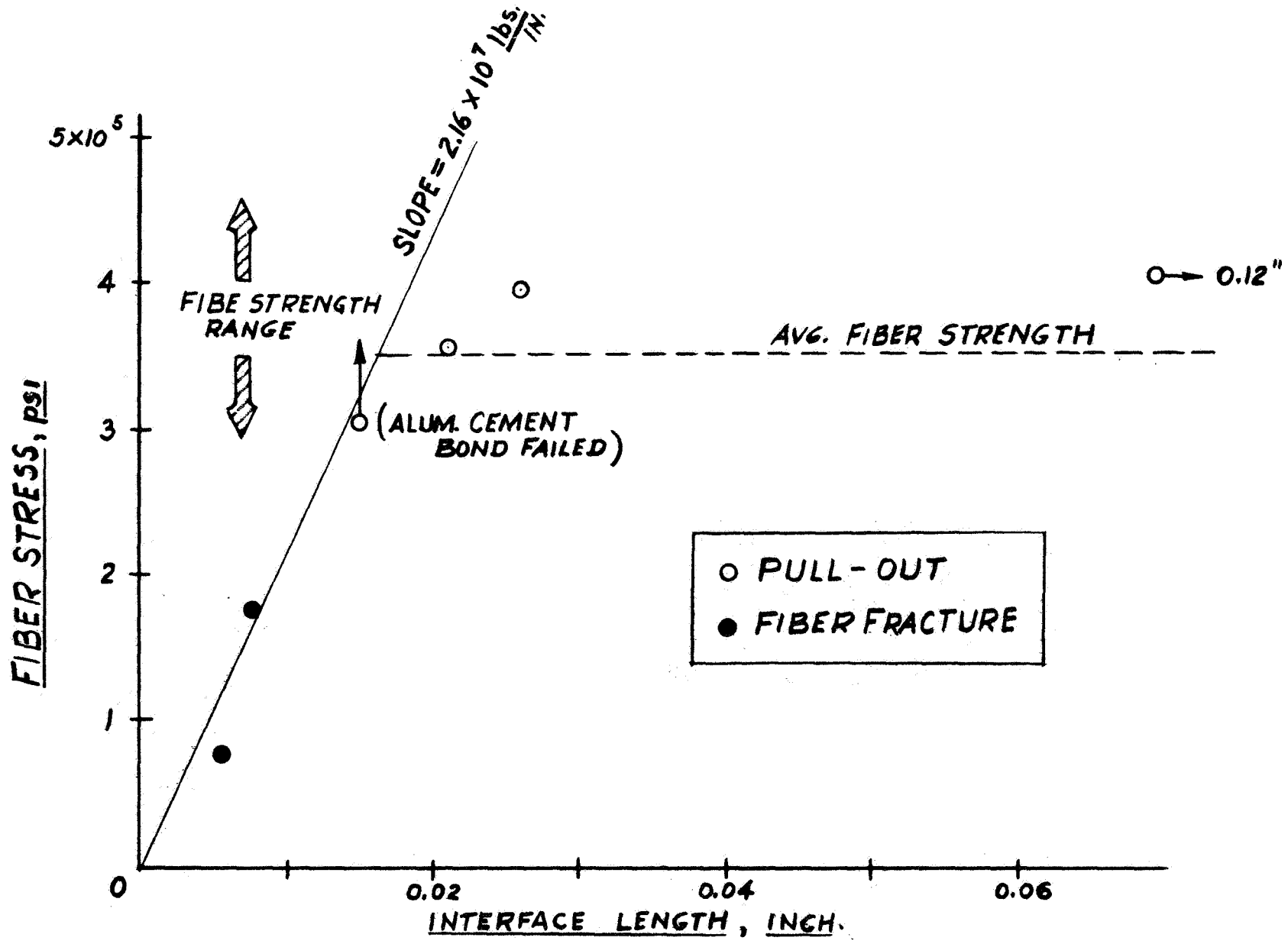


FIG. B6 :- STRENTH OF Al/C BOND